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HOT WATER DRYING OF SUBBITUMINOUS COAL

by  
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Bachelor of Science (Chemical Engineering)  
Regional Engineering College, Rourkela, India

A Thesis

Submitted to the Graduate Faculty

of the

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for the degree of

Master of Science

Grand Forks, North Dakota

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1984

# Hot Water Drying of Subbituminous Coal

Ajit Thomas Joseph

The University of North Dakota, 1984

Faculty Advisor: Dr. Dorab N. Baria

This study investigated the reduction of moisture of a Sarpy Creek Montana subbituminous coal when it was heated under pressure in a water slurry. This method of drying is called hot water drying. The temperature range investigated was from 260 C to 360 C. Two particle sizes were studied, namely, 0.4699 cm and 0.0505 cm mean particle diameters. The average initial moisture content of the coal was 26.87 percent.

The hot water drying was performed in a cold charge autoclave. The coal-water slurry was charged, heated to desired temperature and held at that temperature for 15 minutes residence time. It was then allowed to cool to ambient temperature.

The moisture reduction for the 0.4699 cm diameter particle averaged at 72.52 percent as drying temperature was increased from 260 to 360 C, while for the 0.0505 cm diameter particle the moisture reduction averaged at 77.26 percent for a similar temperature range. The moisture reduction was found to be independent of both the particle size and the drying temperature.

There was significant reduction of sodium content of the coal on hot water drying, and the reduction increased with drying temperature. Sodium reductions of 50 to 75 percent were obtained. Particle size did not have any significant influence on sodium removal. The calorific value of the coal also increased as a function of the drying temperature. The calorific value of the larger hot water dried particles increased from 6098 cal/g dry coal to 6514 cal/g dry coal (average calorific value of the original coal was 5960 cal/g dry coal) as the drying temperature was varied from 260 to 360 C, while the calorific value of the smaller hot water dried coal particles increased from 5544 to 6098 cal/g dry coal (average calorific value of the original coal was 5475 cal/g dry coal). The sulfur removal, though low, was found to decrease with increase in drying temperature for both the particle sizes. Reduction in sulfur content and increase in heating value were higher for the larger particle size.

The equilibrium moisture content of the dried coal, as found by moisture absorption tests, decreased as the drying temperature increased. There was no significant change in the ash content of the coal on hot water drying. Acid groups reduction increased as the drying temperature increased for both the particle sizes. There was no significant effect of particle size on acid group reduction.



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J774

This thesis submitted by Ajit Thomas Joseph in partial fulfillment of the requirements for the Degree of Master of Science from the University of North Dakota is hereby approved by the Faculty Advisory Committee under whom the work has been done.

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This thesis meets the standards for appearance and conforms to the style and format requirements of the Graduate School of the University of North Dakota, and is hereby approved.

A. William Joseph 7/26/84

Title HOT WATER DRYING OF SUBBITUMINOUS COAL

Department Chemical Engineering

Degree Master of Science

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## CONTENTS

LIST OF FIGURES . . . . .	viii
LIST OF TABLES . . . . .	x
ACKNOWLEDGMENTS . . . . .	xii
ABSTRACT . . . . .	xiii

<u>Chapter</u>	<u>page</u>
I. INTRODUCTION . . . . .	1
II. LITERATURE SURVEY . . . . .	6
III. THE HOT WATER DRYING CONCEPT . . . . .	16
IV. EXPERIMENTAL AND EQUIPMENT DETAILS . . . . .	20
Equipment Details . . . . .	20
Coal Studied . . . . .	24
Preparation of the slurry and Loading of the Autoclave Charge . . . . .	25
Autoclave Heating . . . . .	27
Removal of Product from Autoclave . . . . .	29
Removal of Excess Moisture from Hot Water Dried Coal . . . . .	30
Determination of surface moisture upon wetting . . . . .	31
Moisture Reabsorption Tests Using a 100% Humidity Chamber . . . . .	32
Analyses . . . . .	33
Analysis of coal . . . . .	33
Analysis of Filtrate . . . . .	35
Analysis of the Residual Gas . . . . .	36
V. EXPERIMENTAL RUNS . . . . .	37
VI. RESULTS AND DISCUSSION . . . . .	40
Effect of Drying Temperature on Moisture Reduction . . . . .	40
Effect of Drying Temperature on Moisture Reabsorption and the Equilibrium Moisture Content . . . . .	44



Effect of Drying Temperature on Ash content of coal . . . . .	54
Effect of Drying Temperature on Sulfur Removal . . . . .	57
Effect of Drying Temperature on Removal of Inorganic Metals . . . . .	61
Effect of Drying Temperature on Acid Groups Removal . . . . .	75
Effect of Drying Temperature on Calorific Value of Coal . . . . .	82
Overall Material Balance Closure . . . . .	85
Effect of Drying Temperature on Residual Gas Composition . . . . .	86
VII. CONCLUSION AND RECOMMENDATION . . . . .	89
Conclusion . . . . .	89
Recommendation . . . . .	90

<u>Appendix</u>	<u>page</u>
A. DETERMINATION OF MOISTURE AND ASH OF COAL . . . . .	91
Sample Data for Run #8 . . . . .	91
Sample Calculation for Moisture and Ash Contents . . . . .	92
B. CALCULATION OF THE AMOUNT OF MOISTURE INTAKE (ALSO SURFACE MOISTURE) BY ORIGINAL COAL ON WETTING WITH WATER . . . . .	94
Sample Data From Coal Wetting Experiments for 0.4699 cm Mean Diameter Original Coal Particles . . . . .	94
Sample Calculation of Water Intake by 0.4699 cm Mean Diameter Original Coal Particles on Wetting with Water . . . . .	95
C. CALCULATION OF THE INHERENT MOISTURE CONTENT OF HOT WATER DRIED COAL AND THE PERCENT MOISTURE REMOVAL ON HOT WATER DRYING . . . . .	98
Sample calculation of the Inherent Moisture Content of the of the Hot Water Dried Coal . . . . .	98
Sample Data for Run #8 . . . . .	98
Calculation of the Inherent Moisture Content . . . . .	98
Sample Data for Run #8 . . . . .	99
Sample Calculation for Moisture Removal . . . . .	99

D.	MOISTURE REABSORPTION AND DETERMINATION OF EQUILIBRIUM MOISTURE CONTENT OF ORIGINAL AND HOT WATER COALS USING A 100 % HUMIDITY CHAMBER . . . . .	100
E.	TOTAL MATERIAL BALANCE . . . . .	105
	Sample Data for Run #10 . . . . .	105
	Sample Calculation for Mass of Residual Gas Formed . . . . .	106
	Sample Total Material Balance Closure . . . . .	107
F.	CALCULATION FOR THE SULFUR CONTENT . . . . .	108
	Sample Data for Run #18 . . . . .	108
G.	MATERIAL BALANCE CLOSURE, REMOVAL AND NORMALIZATION FOR SULFUR . . . . .	110
	Sample Data for Run #5 . . . . .	110
	Sample Material Balance Closure for Sulfur . . . . .	111
	Sample Calculation for Sulfur Removal . . . . .	111
	Normalization for Sulfur . . . . .	112
H.	CALCULATION OF SODIUM, POTASSIUM, MAGNESIUM, AND CALCIUM CONTENTS OF THE COAL AND THE FILTRATE . . . . .	113
	Sample Data for Run #19 . . . . .	113
	Sample Calculation for the Metals Content . . . . .	114
	Sample Calculation of the Metal Oxides Content in the Ash . . . . .	115
	Sample Data for the Metals Content of the Filtrate . . . . .	116
	Sample Calculation for the Metals Content of the Filtrate . . . . .	116
I.	MATERIAL BALANCE CLOSURE, REDUCTION AND NORMALIZATION FOR METALS . . . . .	118
	Sample Data for Run #11 . . . . .	118
	Sample Calculation for Material Balance Closure for Sodium . . . . .	119
	Sample Calculation for the Removal of Sodium . . . . .	120
	Normalization for Sodium . . . . .	120
	Sample Material Balance Closure for Potassium . . . . .	121
	Sample Calculation for the Removal of Potassium . . . . .	121
	Normalization for Potassium . . . . .	122
	Sample Data for Magnesium . . . . .	122
	Sample Calculation for the Removal of Magnesium . . . . .	123
	Normalization for Magnesium . . . . .	123



	Sample Data for Calcium . . . . .	124
	Sample Calculation for the Removal of Calcium . . . . .	124
	Normalization for Calcium . . . . .	125
J.	DETERMINATION OF TOTAL ACID GROUPS CONTENT IN COAL AND REDUCTION IN ACID GROUPS CONTENT ON HOT WATER DRYING . . . . .	126
	Sample Data for Run #8 . . . . .	126
	Sample Calculation for Acid Groups Contents . . . . .	127
	Sample Calculation for Reduction in Acid Groups Content . . . . .	129
K.	CARBON DIOXIDE CONTENT OF THE RESIDUAL GAS . . . . .	130
	Sample Data for run #11 . . . . .	130
	Sample Calculation for Carbon Dioxide Content of Residual Gas . . . . .	131
L.	CALORIFIC VALUE OF COAL . . . . .	132
	Sample Data for Run #5 . . . . .	132
	Corrections to be Made . . . . .	133
	Sample Calculation for the Calorific Value . . . . .	134
M.	ANALYSIS OF THE AS RECIEVED COAL . . . . .	136
N.	STATISTICAL ANALYSIS . . . . .	138
	Test on the Difference of The Two Mean Percent Moisture Reductions . . . . .	138
	Test on the Mean Percent Ash of the Hot Water Dried Coal . . . . .	141
	Test on the Mean Percent Sulfur Removal . . . . .	144
	REFERENCES . . . . .	148

## LIST OF FIGURES

<u>Figure</u>	<u>page</u>
1. Sketch of Autoclave and its Internals (29) . . . . .	21
2. Sketch of the Piping for the Autoclave (29) . . . . .	22
3. Temperature Histories of Autoclave Inside and Heating Block . . . . .	28
4. Moisture Reduction as a Function of Drying Temperature . . . . .	43
5. Moisture Reabsorption by Original Coal (0.4669 cm Mean Diameter Particles) used for Run #5 in 100% Humidity Chamber . . . . .	47
6. Moisture Reabsorption by Hot Water Dried Coal (0.4669 cm Mean Diameter Particles) in 100% Humidity Chamber . . . . .	48
7. Moisture Reabsorption By Original Coal (0.0505 cm Mean Diameter Particles) used for run #18 in 100% Humidity Chamber . . . . .	49
8. Moisture Reabsorption by Hot Water Dried Coal (0.0505 cm Mean Diameter Particles) in 100% Humidity Chamber . . . . .	50
9. Equilibrium Moisture Content of Hot Water Dried Coal as a Function of Drying Temperature . . . . .	53
10. Ash Content (dry basis) of Hot Water Dried Coal as a Function of Drying Temperature . . . . .	56
11. Sulfur Reduction (normalised) as a Function of Drying Temperature . . . . .	60
12. Sodium Reduction (normalised) as a Function of Drying Temperature . . . . .	71
13. Potassium Reduction (normalised) as a Function of Drying Temperature . . . . .	72

14.	Magnesium Reduction (normalised) as a Function of Drying Temperature . . . . .	73
15.	Calcium Reduction (normalised) as a Function of Drying Temperature . . . . .	74
16.	Reduction in Acid Groups Content as a Function of Drying Temperature . . . . .	76
17.	Acid Groups Content of Hot Water Dried Coal as a Function of Drying Temperature . . . . .	77
18.	Carbon Dioxide Content in Residual Gas as a Function of Drying Temperature . . . . .	80
19.	Calorific Value of Hot Water Dried Coal as a Function of Drying Temperature . . . . .	84
20.	Moisture Intake by As Recieved Coal Particles on Wetting With Water . . . . .	97
21.	Moisture Reabsorption by CAR (0.4669 cm Mean Diameter Particles) in 100% Humidity Chamber .	103
22.	Moisture Reabsorption by CAR (0.0505 cm Mean Diameter Particles) in 100% Humidity Chamber .	104



## LIST OF TABLES

<u>Table</u>	<u>page</u>
1. Average Proximate and Ultimate Analysis of the Original Coal . . . . .	25
2. Details of the Experimental Runs . . . . .	38
3. Grouping of Coal for the Various Runs . . . . .	39
4. Moisture Reduction and Inherent Moisture Contents of Original Coal and Hot Water Dried Coal . . . . .	42
5. Moisture Reabsorption Tests on 0.4699 cm Mean Particle Diameter HWD Coal in 100 % Humidity Chamber . . . . .	45
6. Moisture Reabsorption Tests on 0.0505 cm Mean Particle Diameter HWD Coal in 100 % Humidity Chamber . . . . .	46
7. Equilibrium Moisture Content of HWD Coal and CAR . . . . .	52
8. Ash Content of CAR and HWD Coal . . . . .	55
9. Material Balance Closures for Sulfur . . . . .	58
10. Sulfur Removal on Hot Water Drying . . . . .	59
11. Material Balance Closure for Sodium . . . . .	62
12. Material Balance Closure for Potassium . . . . .	63
13. Material Balance Closure for Magnesium . . . . .	64
14. Material Balance Closure for Calcium . . . . .	65
15. Metal Oxides Content in ash of Original Coal and HWD Coal . . . . .	66
16. Sodium Removal on Hot Water Drying . . . . .	67
17. Potassium Removal on Hot Water Drying . . . . .	68

18.	Magnesium Removal on Hot Water Drying . . . . .	69
19.	Calcium Removal on Hot Water Drying . . . . .	70
20.	Reduction of Acid Groups Content on Hot Water Drying . . . . .	75
21.	Carbon Dioxide Content in Residual Gas . . . . .	79
22.	Calorific Value of CAR and HWD Coal . . . . .	83
23.	Overall Material Balance . . . . .	86
24.	Residual Gas Composition (normalised, air free) . .	87
25.	Moisture Intake on Wetting CAR . . . . .	96
26.	Moisture Reabsorption Tests on CAR (both 0.4669 cm and 0.0505 cm Mean Diameter Particles) in 100% Humidity Chamber . . . . .	102
27.	Analysis of CAR . . . . .	136
28.	Analysis of CAR . . . . .	137



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## ABSTRACT

This study investigated the reduction of moisture of a Sarpy Creek Montana subbituminous coal when it was heated under pressure in a water slurry. This method of drying is called hot water drying. The temperature range investigated was from 260 C to 360 C. Two particle sizes were studied, namely, 0.4699 cm and 0.0505 cm mean particle diameters. The average initial moisture content of the coal was 26.87 percent.

The hot water drying was performed in a cold charge autoclave. The coal-water slurry was charged, heated to desired temperature and held at that temperature for 15 minutes residence time. It was then allowed to cool to ambient temperature.

The moisture reduction for the 0.4699 cm diameter particle averaged at 72.52 percent as drying temperature was increased from 260 to 360 C, while for the 0.0505 cm diameter particle the moisture reduction averaged at 77.26 percent for a similar temperature range. The moisture reduction was found to be independent of both the particle size and the drying temperature.

There was significant reduction of sodium content of the coal on hot water drying, and the reduction increased with drying temperature. Sodium reductions of 50 to 75 percent were obtained. Particle size did not have any significant influence on sodium removal. The calorific value of the coal also increased as a function of the drying temperature. The calorific value of the larger hot water dried particles increased from 6098 cal/g dry coal to 6514 cal/g dry coal (average calorific value of the original coal was 5960 cal/g dry coal) as the drying temperature was varied from 260 to 360 C, while the calorific value of the smaller hot water dried coal particles increased from 5544 to 6098 cal/g dry coal (average calorific value of the original coal was 5475 cal/g dry coal). The sulfur removal, though low, was found to decrease with increase in drying temperature for both the particle sizes. Reduction in sulfur content and increase in heating value were higher for the larger particle size.

The equilibrium moisture content of the dried coal, as found by moisture absorption tests, decreased as the drying temperature increased. There was no significant change in the ash content of the coal on hot water drying. Acid groups reduction increased as the drying temperature increased for both the particle sizes. There was no significant effect of particle size on acid group reduction.



## Chapter I

### INTRODUCTION

At the present rate of consumption the world's economically recoverable crude oil and natural gas supply will soon be running out. The peak production level is expected to occur by the year 2000. Oil and natural gas reserves are likely to last for another 60 to 90 years (1,2)<sup>1</sup>. Hence unless some other forms of energy are also developed to supplement or even replace oil and gas, there could be a drastic change in the way of life people are used to in the United States (1,2).

Coal, especially low-rank western coal, is an excellent alternate source of energy in the U.S. There are enormous deposits (over 1 trillion tons of identified resources) of low-rank coals in the U.S., and their extraction costs are cheap relative to the high rank eastern coals (between 50 to 75 cents per million Btu) (3). Major deposits of low-rank coals are also found in many other countries, most notably the USSR, Australia, Canada, and the central and eastern European nations (4).

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<sup>1</sup>Numbers in paranthesis that are underlined refer to references cited at the end of this report.

Low-rank coals can be characterized as high in moisture (20 to 40 percent), low in sulfur, and variable in sodium content. Low-rank coals can be utilized to generate electricity, and to produce synthetic or substitute natural gas (SNG) by gasification and synthetic liquid fuels by liquefaction.

In the years ahead, large quantities of coal will have to be transported to other places for utilization due to economic and/or environmental factors. In certain areas, slurry pipeline transport appears to have an added advantage over the conventional methods of transport. The conventional methods of transport are limited by a number of factors, like high transportation costs, rail car availability, urban congestion, scheduling, etc. (3). Also, the potential markets for low-rank coals can be enhanced if slurry pipeline transport is considered. This method, as an alternative to rail shipment, has been considered for high-rank coals for a number of years. Slurry pipeline transportation of low-rank coals has a few disadvantages; two of which are the limited supply of water in areas where these coals are located and the high inherent moisture content of the low-rank coals (3).

The high moisture content in low-rank coals is partly trapped in pores and capillaries, and partly bound in the molecular structure. Application of heat is the only prac-



tical means for removing this inherent moisture (4). The high inherent moisture content lowers the heating value of the slurry, resulting in the need for larger pipe diameter, more water, and higher pumping costs to deliver same unit of heating value in the slurry as compared to high-rank, low moisture coals. The high inherent moisture in low-rank coal slurries decreases the heating value available for direct combustion and gasification processes and, in fact, precludes their use in these processes without moisture reduction.

There is currently much interest in direct combustion of 60 to 70 percent solid loaded high-rank coal slurries as replacement fuel in oil-fired boilers. These slurries can also be used as feed to the Texaco gasifier (3).

If the water requirements for slurring coal can be reduced, the potential of long distance coal slurry transportation and/or direct utilization of slurry in combustion or gasification for low-rank coals could be greatly enhanced. The process called 'hot water drying' has shown that this reduction in water requirements may be possible (3). This process basically converts the inherent moisture to part of the carrier medium.

Hot water drying coal also greatly reduces sodium (present as sodium salt of carboxylic acids in the coal) and to a lesser extent potassium, calcium and magnesium (present as

salts of carboxylic acids) and sulfur. Sodium in coal causes ash fouling in boilers and other similar equipment, and is therefore an undesirable constituent. Low-rank coals are normally low sulfur coals and hence sulfur reduction on hot water drying is also low (4). Low sulfur content reduces the cost of compliance with emissions standards (4). The heating value of the hot water dried coal is higher than that of the original coal, therefore improving the economics of the hot water drying process.

Prior to 1970, production of low-rank coals was only 1 to 2 percent of the total annual U.S coal production. Utilization of these resources was important only in local areas and was on a small scale. Production of low-rank coals mushroomed during the 1970's, accounting for nearly all of the growth in coal production achieved during that decade. In 1980, low-rank coals accounted for nearly 24 percent of the total U.S coal production, and are poised for further growth. By 1990, these coals may make up for 40 to 50 percent of a greatly expanded total (4).

A few experimental coal gasification units employing the Texaco gasifier have been successfully established in this country and elsewhere. Many more are likely to be constructed in the future. Two of the existing plants are the 150 metric tons/day (t/d) plant in Oberhausen in the Federal Republic of Germany and the 13.6 t/d pilot plant in Monte-

bella, California (5). The Texaco gasification process has an inherent disadvantage in that it cannot gasify efficiently the high moisture content low-rank coals. North American lignites contain from 30 to 40 percent inherent moisture, which makes the coal-water slurry difficult to gasify. Hence, if part of the inherent moisture is driven out of the coal while in a water slurry, the resulting coal-water slurry would contain less water, making the gasification process easier.

In this study the effect of drying temperature and particle size on moisture reduction of a Sarpy Creek Montana subbituminous coal was investigated. The coal was dried as a slurry with water in a cold charge autoclave at high pressures. The slurry concentration used was 1:1 (liquid to solid mass ratio), and residence time was held at 15 minutes. The effect of drying temperature and particle size on ash, carboxylic acid groups, sulfur, sodium, calcium, potassium, magnesium contents and the gross heating value of the subbituminous coal were also studied. Humidity chamber experiments were performed to determine the effect of drying temperature and particle size on the equilibrium moisture content of the coal.



## Chapter II

### LITERATURE SURVEY

Conventional drying exposes coal to a hot stream of combustion gases to drive off moisture by heating the coal, resulting in a hot dried product (4). Typical processes used in drying coal are the Perry, roto louvre, fluid bed, pneumatic, and belt or tray dryers. Most of these processes may be used in drying coal before transportation to power plants. These dryers employ an inert atmosphere and do not produce the desirable stable dried product. The unstable dried coals from these dryers exhibit a pronounced slacking tendency, and thus any handling of the dried coal reduces the particle size and creates dust. The extent of this problem is related to the degree of drying, the original size of the particle, and possibly the petrographic structure of the coal (4).

Fleissner in the 1920's tried to overcome this problem by producing stable dried coal. He did this by drying under pressure with saturated steam (6,7). Fleissner's pioneering work resulted in growing interest in the area of steam drying of coals all over the world. Stanmore, Baria, and Paulson (8) have reviewed the steam drying processes for low-rank coals that have resulted from Fleissner's initial work

on steam drying. A few Fleissner's steam drying plants were built in Europe to dry European brown coals and some are still in operation today.

Fleissner or similar processes (9,10,11,12,13,14) have been used to steam dry North Dakota lignites and extensive studies have been carried out in that direction. Lavin, Gauger, and Mann (9) produced a coal with good weathering properties, the moisture content of which was reduced from 36 to 16 percent. Cooley and Lavine (10) found that the slacking, shattering, and burning properties of lignite dehydrated in oil-steam mixtures surpassed the properties of lignite dried using only steam. Effect of the process temperature on the amount of water removed from the lignite was studied by Harrington, Parry, and Koth (11). They determined that the extent of water removal from smaller lumps was greater than that from larger lumps.

In 1955 Oppelt, Ellman, and Onstad (12) found that the moisture removal was a function of the lignite structure and the processing temperature. The moisture reduction was found to increase with increase in temperature. Also the extent of water removed from smaller lumps was greater than that from larger lumps, thus confirming the work of Harrington, Parry, and Koth (11). Four years later, Oppelt, Kube, and Kamps (13) showed that the temperature had a significant effect on the amount of water removed from the lignite in



the pressure range from 27 to 102 atm. More recently Sapatnekar (14) found that for particle size of 3.15 cm the moisture reduction increased from 45 to 73 percent for steam drying temperatures from 250 to 270 C.

The Fleissner process, using saturated steam at elevated temperatures and pressures, has been used to steam dry Australian brown coals to improve the coal quality. Bainbridge and Satchwell (15) showed a reduction in moisture content from 67 to 10.7 percent and from 49 to 27 percent for coals from Yallourn and Latrobe seams respectively. Stanmore and Boyd (16) tested experimentally a mathematical model for heat transfer within and water removal from a long radially shrinking coal cylinder. They found out that moisture removal is a function of the interior temperature of the particle. Once the interior temperature equalled the surface temperature, the moisture removal was independent of residence time. Hence, the moisture removal during steam drying is a heat transfer limited process, limited by heat conduction within the particle.

Koppelman (17,18,19) patented a process to dewater lignite at high temperatures in a water slurry using steam. The dried coal contained between 1 to 2 percent moisture and heating value were increased from 4000 cal/g to 7000 cal/g (20). The process also claimed to decrease the sulfur and oxygen content of the coal (21).

Moisture adsorption and desorption tests on coal were carried out to gain insight into how the water is bound to the coal or carbon and to observe the behavior of the solid material as water is added or removed. A hysteresis effect was observed by Gordon, Lavine, and Harrington (22) on drying curves for freshly mined and air dried lignites. The hysteresis effect almost disappeared after the lignites were steam dried. Lignites dried in controlled humidity conditions gave different results. From their experiments on controlled humidity drying of lignite, Gordon and others (22) concluded that the moisture in lignite must be considered as part of its physical structure. This conclusion was partly based on the fact that they obtained an inferior lignite as their product.

Kube (23) found out that as drying temperature increased, the steam dried and other thermally treated lignite regained less moisture. He studied the adsorption and desorption of water vapor at 40 C by freshly mined, thermally treated, and steam dried lignites. He found out that the hysteresis effect present in the drying curves for freshly mined lignites, also called the desorption-adsorption hysteresis loop, disappeared after drying or thermal heating. He noticed no hysteresis in moisture retention when lignites thermally treated at 370 C were taken through three complete adsorption-desorption cycles. He also found that some of the weight gain of the lignites was due to oxygen sorption.



The steam drying process has been modified by drying the coal in a slurry with water at high temperature under pressure. This method is especially advantageous when using low-rank coals since it produces a dried coal in a slurry containing less inherent moisture than the original coal. Evans and Siemon (24) were the first to show that about 40 % of the moisture in Australian Yallourn brown coal could be removed by drying it at 109 C under pressure with nitrogen in either water or mineral oil. They showed that the percent moisture reduction in the coal greatly depended on the temperature, and was independent of the pressure and the fluid medium, provided the coal was removed from the liquid medium before cooling or depressurising. They worked with pressures upto 5 times the saturation pressure of water at 109 C.

A North Dakota lignite was upgraded by Elliot (25) by heating it in a water slurry at a temperature of 320 C for 1 hr. A decrease in coal mass of 21.5 percent was observed. A low final moisture content of approximately 2 percent was obtained and the heating value was increased from 5,200 to 6,300 cal/g. Also, total sulfur was decreased by approximately 10 percent and total ash mass was decreased by 16 percent. The resulting product has apparent physical characteristics very similar to the feed material. The product is recovered as a solid powder and there is no oily or greasy texture to the material. According to Elliott, water



was necessary to gain the added benefit of ash removal by leaching.

Murray and Evans (26) studied the drying of Yallourn brown coal from Australia in water at temperatures between 150 to 300 C. They concluded that the liquid water removal was initiated by a disruption of the coal/water interactions induced by the thermal destruction of the oxygen functional groups, and confirmed that the carbon dioxide evolution, changes in surface wettability and coal shrinkage were responsible for the expulsion of water. Alkali and alkaline earth metals, chlorine, and sulfur were also partly removed by the process, whereas iron, aluminium, and silicon were barely affected.

Murray and Evans (26), also showed that the decomposition of the phenolic groups commenced at 150 C and it was quite marked above 200 C. Alcoholic groups were stable upto 200 C, but decomposed readily thereafter. The main product of decomposition of both these groups is water. Their studies showed that free carboxylic acid groups were stable up to 150 C, but decompose readily at higher temperatures. There was a small but steady breakup of carboxylate groups from 20 to 200 C, with increased decomposition at temperatures higher than 200 C. They concluded that both these groups yield carbon dioxide, and the amount of carbon dioxide predicted to be evolved due to breakup of these groups account for

about 4/5th of the amount of CO<sub>2</sub> actually recovered. Carbonyl groups were stable up to 150 C, but decomposed at higher temperatures, possibly leading to the formation of carbon monoxide, and probably some carbon dioxide. Sodium removal increased with temperature while the removal of calcium increased with temperature above 200 C.

Using nuclear magnetic resonance (nmr) techniques, Lynch and Webster (27) found a rapid reduction in the coal/water interaction (or affinity for each other) at drying temperatures greater than 150 C when coal was dewatered in a water slurry between 70 and 340 C using the methods adopted by Murray and Evans (26). They were able to make a distinction between water which interacts with the coal and free moisture using nmr techniques. They checked for frozen or unbound water in the dried coal using proton nmr (Hnmr) after cooling to 0 C. The unbound and non-frozen (or bound) moisture was found to decrease for the dried product.

Desorption of moisture from Yallourn coal was studied by Evans (28). Run-of-mine Yallourn coal was dried by equilibrating in atmosphere with various relative humidities. He observed various amounts of contraction in the coal structure as moisture was removed from the coal progressively. Little contraction was observed from coal cylinders as the bulk water was lost. In the second stage of water removal, capillaries of diameter 120 nm down to 5 nm tended to col-



lapse. Next, in the third stage, the multilayer sorbed water was removed, and the shrinkages were large and disproportionate. These shrinkages occurred due to strengthening of the hydrogen bond bridges between micelles. In the last stage, when the water content falls below two water layers around each micelle, further loss entails the formation of less stable linkages between micelles, and the shrinkage drops to zero and may eventually become negative, i.e., swelling may occur with the development of further porosity. The final dried coal cylinders have only 55 % of their original volume, but have an empty porosity of 50 %.

Recently Maas (29) studied the hot water drying of a North Dakota lignite from Larson at temperatures varying from from 200 to 285 C in a cold charge autoclave at saturation pressures. His results indicated that the moisture removal, which varied from 25 to 75 percent, was a linear function of temperature, and that slurry concentration did not significantly affect moisture reduction. Maas noticed that the equilibrium moisture content of the hot water dried coal decreased as the drying temperature increased. He also found that as the drying temperature increased carbon dioxide formation, gross heating value, and acid group decomposition increased. Ash content of the coal did not change significantly but the sodium content of the lignite was reduced considerably during hot water drying. He also determined an empirical equation to calculate the final moisture content of the coal studied after hot water drying.



Baria, Maas, Paulson (3) studied the hot water drying of a Velva and Indian Head lignite from North Dakota at temperatures ranging from 200 to 340 C in a hot charge autoclave. Their studies indicated that it took less time for smaller particles to reach a constant moisture content upon hot water drying. This is consistent with the work of Stanmore and Boyd (16) who indicated that the extent of moisture reduction was a function of the interior temperature of the coal particle. The moisture reduction increased and equilibrium moisture decreased as the drying temperature increased. The carboxylic acid groups started to decompose at temperature above 240 C and heating value of the coal increased with drying temperature behind 240 C. The extent of sodium removal also increased with temperature. No significant amount of calcium or sulfur was removed. They showed that a highly loaded as recieved coal-water slurry of a low-rank coal which would be difficult or impossible to pump, would become pumpable after hot water drying.

A continuous process development unit has been constructed for hot water drying of 200 lb/hr of a 50 % solid coal-water slurry at the University of North Dakota Energy Research Center (3). This unit will produce enough hot water dried coal slurry to carry out large scale combustion tests of coal-water slurry and to perform rheological studies.

Since 1978 Saskatchewan Power Corporation has undertaken considerable experimental, engineering and economic work to develop a low-cost, low-energy process for dewatering high moisture content fuels such as Saskatchewan lignites (30). The process presently adopted at the Saskatchewan Power Corporation consists of contacting finely divided lignite with saturated steam at temperatures in the range of 250 to 350 C between residence times 5 and 90 minutes. The product from the process was non-friable, dust free, and not subject to spontaneous combustion. High equilibrium moisture reductions have been observed, from 35 percent down to 15 percent. During the process considerable decrease in alkali metals, such as sodium, potassium and sulfur have been noticed. The levels of the alkali metals and sulfur decreased by at least 50 percent due to the process. Future work will centre on process optimization using batch and small continuous units, using a process with non-saturated steam instead of saturated steam.

## Chapter III

### THE HOT WATER DRYING CONCEPT

Steam drying or Fleissner drying process was developed to dry low-rank coals and overcome the disadvantages of the conventional drying processes, such as, Perry, roto louvre, pneumatic, fluid bed, and belt or tray dryers. Hot water drying process is more recent modification of the steam drying process.

Steam drying is a process which utilizes saturated steam under pressure to remove water as a liquid from coal. This process causes the coal particles to shrink in size but retain their lump form. The product is reduced in moisture and has improved handling and weathering properties (8). Hot water drying is also a process for the removal of water in liquid phase from coal by heating a coal-water slurry under pressure. The drying temperatures are sufficiently high so that some oxygen functional groups in the coal break up to form carbon dioxide (26). Klein (31) proposed the following mechanism for removal of water in steam drying process :

1. Water in a liquid phase is pushed out of the pores by the carbon dioxide which forms due to the decomposition of the oxygen functional groups.



2. As pores contract and the coal shrinks, surplus water is expelled.
3. The differences in thermal expansion of coal and water causes water to be forced from the pores during heating.
4. Coal is less able to bind water because of surface modifications which reduces the ability of the coal surface to bind water. This phenomena is due to the replacement of the hydrophillic carboxyl groups on the surface of the coal by the hydrophobic hydrocarbon groups. They tend to repel the liquid water from the surface and also prevent water from reentering the pores once it has been driven out into the carrier medium.
5. At higher temperatures, viscosity of water is reduced, hence allowing water to run freely from the coal.

The first three mechanisms are mainly responsible for the expulsion of water from the coal particle. The second and third mechanisms dominate at lower drying temperatures and the first is predominant at higher temperatures.

Energy requirements for drying are minimised for steam and hot water drying processes because the water is removed as a liquid rather than as a vapor. The hot water drying process is well suited for heat recovery (3). Metal ions, such as sodium, potassium, magnesium, and calcium, in low

rank coals are attached to the coal structure through the carboxylic groups, and are released upon the breakup of the carboxylic groups, and diffuse out into the aqueous phase. Slurries made with some hot water dried coals have been reported to be stable with little or no settling (3). This stability of the slurry is important when pipeline transportation of coal is considered.

The hot water drying process is easily operated on a continuous basis using slurry pumps to move the coal-water slurry into and out of the pressurised drying units, while the steam drying process which handles coal lumps has to use lock hoppers for charging and discharging coal into and out of pressurised dryers. There is little or no shrinkage of the coal upon hot water drying, while there is some shrinkage of coal and inherent dust problems associated with steam drying.

Although removal of liquid water from coal has a marked dependence on the temperature reached, it does not depend on the pressure and the fluid medium (26). This is valid only if the hot water dried coal is removed from the liquid medium before cooling or depressurising. When the hot water dried coal is allowed to cool in the slurry it reabsorbs part of the expelled water depending on the processing temperature. The higher the drying temperature, the lower is the amount of water reabsorbed. Depending on the coal, at

low processing temperatures almost all the the expelled water might be reabsorbed, whereas at higher temperatures there might be no reabsorption of water at all (26).



## Chapter IV

### EXPERIMENTAL AND EQUIPMENT DETAILS

#### EQUIPMENT DETAILS

The hot water drying of coal was conducted in an autoclave manufactured by Autoclave Engineers. The material of construction of the autoclave was 316 stainless steel. This material was corrosion resistant and withstands high temperatures. The autoclave was rated at 750 atm at 22 C and the nominal capacity was 500 ml. The maximum torque for the set screws was from 20 to 23 Newton-meters (180 to 204 in-lbs). The sketch of the autoclave is shown in Figure 1.

A sketch of the piping assembly for the autoclave is shown in Figure 2. It consisted of gas sampling valve, thermocouple, pressure gauge, and a blowout valve. A 110 mm length of tubing was attached permanently to the top cover of the autoclave. The piping assembly was screwed into this length of tubing during an experimental run.

A 2 mm thick 316 stainless steel bucket held the coal-water slurry. This bucket fitted well inside the autoclave with negligible clearance between the bucket and the inner walls of the autoclave. To prevent the bucket from sticking

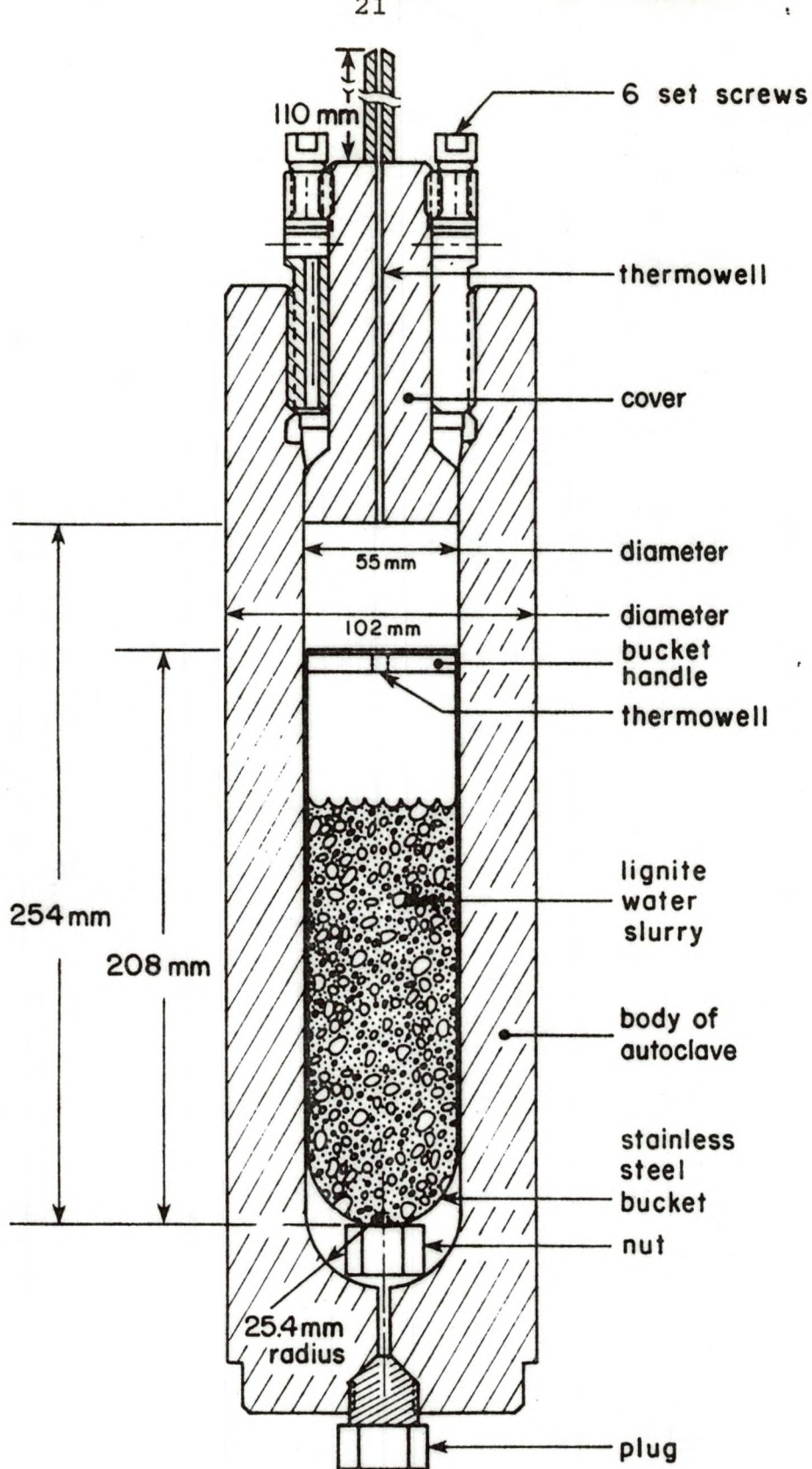


Figure 1: Sketch of the Autoclave and its Internals (29)

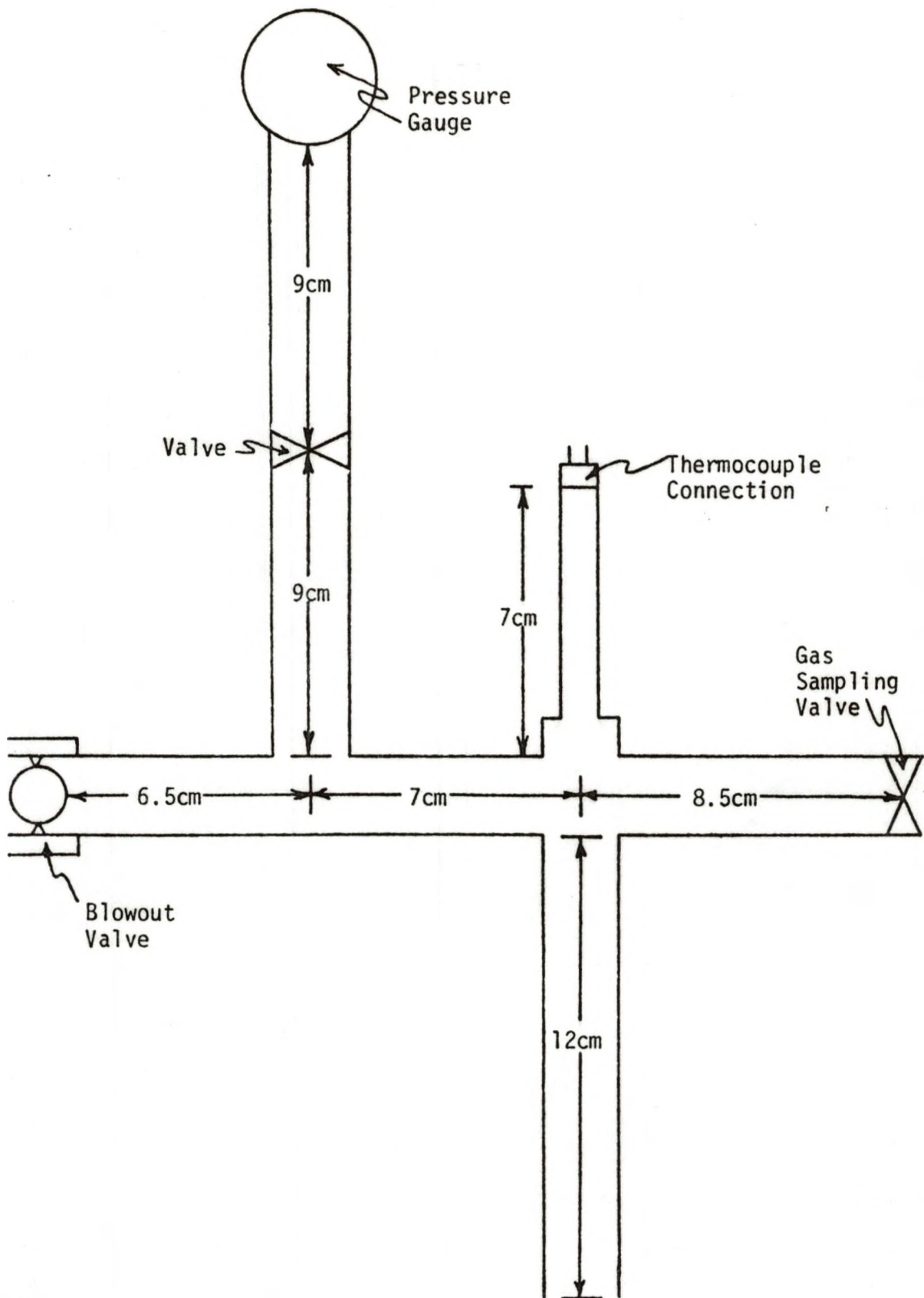


Figure 2: Sketch of the Piping for the Autoclave (29)



to the bottom of the autoclave and to facilitate the removal of the bucket from the autoclave after each run, a nut was placed on the bottom of the autoclave and the bucket rests on it. The bucket was 20.8 cm long and had an outside diameter of 15 cm. A 9 mm stainless steel pipe section, welded onto the bucket, served as the bucket handle. To facilitate the placing of the thermocouple into the slurry, a 6 mm diameter hole was drilled through the center of the bucket.

Six aluminium blocks, each 17.8 cm X 17.8 cm X 3.8 cm in size, surrounded the lower 2/3rd of the autoclave body. The upper 1/3rd of the autoclave body was insulated by a 1.27 cm thick glass wool during a run. This reduced heat loss from the upper portion of the autoclave. The aluminium blocks were surrounded by gypsum insulation 3.8 cm thick. Three electric bead heaters at the bottom of the blocks supplied the heat to the autoclave. There were two thermocouples attached to the autoclave assembly, one for measuring the temperature of the heating blocks and the other for measuring the temperature inside the autoclave. The temperature of the heating block was controlled by a Brown Electric Pyrometer-Temperature Controller. This pyrometer controller was connected to a chromel-alumel thermocouple which was inserted into the blocks before every run. An iron-constantan thermocouple connected to a Leeds and Northrup temperature potentiometer measured the temperature inside the autoclave.

The pressure gauge measured the pressure inside the autoclave. It was capable of measuring upto 690 N/sq. cm. An insulated heating element (not shown in Figures 1 and 2) was wrapped around the pipe to keep the tubing hot enough to prevent any condensation inside the tubes.

#### COAL STUDIED

The coal investigated was Sarpy Creek subbituminous coal from Montana. The coal, contained in a large, properly sealed plastic bag, was placed in a large drum. The drum was also sealed. This arrangement ensured least contact of coal with air. Two particle sizes of the coal, 0.4699 cm (-4 by +4 U.S. Standard mesh) and 0.0505 cm (-30 by +40 U.S. Standard mesh) mean particle diameters, were used in the study. The 'as recieved coal' for a particular particle size refers to the raw coal of that particle size used for the experimental run. This is also refered to as 'original coal'. Before each run, a small, well sampled portion of the original coal was put away for all the analyses.

The average proximate and ultimate analysis of the original coal is given in Table 1. The analysis is expected to vary between individual samples of varing particle sizes as well as samples of the same particle size due to the heterogeneous nature of the coal.

TABLE 1

Average Proximate and Ultimate Analysis of the Original Coal

-----	
Proximate Analysis, percent :	
Volatile Matter	31.80
Moisture	20.90
Fixed Carbon	36.72
Ash	10.58
-----	
Ultimate Analysis, percent (Moisture Free Basis) :	
Carbon	64.77
Hydrogen	5.33
Nitrogen	1.01
Sulfur	0.47
Oxygen (by difference)	15.28
Ash	13.14
-----	

PREPARATION OF THE SLURRY AND LOADING OF THE AUTOCLAVE CHARGE

The autoclave charge, or feed, was a coal-water slurry having a liquid to solid mass ratio of 1:1. This concentration was maintained constant for all runs for both particle sizes.

The raw coal was sieved to obtain the two particle sizes, 0.4699 cm and 0.0505 cm mean particle diameters, to be used in the experimental runs. Each run required 180 g of coal. 30 g of the sieved coal was put in an air tight plastic bottle and kept aside for analysis. The remainder 150 g of the sieved coal was put in a 1000 ml beaker and mixed with 155 g of distilled water.



Maas (29) determined from blank runs, using the same autoclave as this study, that 10 g of water from the slurry was vaporised and lost due to condensation in the piping and fitting in every run. In order to compensate for this loss of water, 5 extra grams of water were added over and above the 150 g of water needed to make a slurry containing 1:1 liquid to solid mass ratio. This gives the slurry a slightly lower solids concentration at the beginning of each run and a slightly higher solids concentration at the end of the run. The mixture was well agitated with a glass rod until a well mixed, homogeneous slurry was obtained. This slurry was then transferred into the steel bucket. The slurry was then ready to be hot water dried in the autoclave.

The steel bucket containing the slurry was lowered inside the autoclave with the help of a wire and allowed to rest on the nut located inside on the bottom of the autoclave. The top cover was closed and all the required nuts and bolts were tightened. The external piping was then attached to the autoclave. The iron-constantan thermocouple was inserted inside the autoclave to measure the temperature inside the autoclave and the chromel-alumel thermocouple was inserted inside the heating block to measure the heating block temperature. The autoclave was now ready to be heated.

AUTOCLAVE HEATING

The power to the autoclave heating block was turned on. The heating block temperature indicator-controller (T-I-C) was also turned on simultaneously. The T-I-C was set at 460 C. The pressure, heating block temperature, and millivolt (mV) reading of the potentiometer corresponding to the temperature inside the autoclave were recorded at regular intervals of 30 min for the first 90 min, then 15 min interval for the next 30 min, then at 5 min interval for the next 60 mins and finally at 30 mins interval for the next 120 mins. The heating time was approximately 5 hrs (300 min) for every run. A heating period of 5 hr was chosen because, the temperature histories of the autoclave interior and heating block become no longer important after 5 hrs. The peak temperatures are the most important temperatures in this study, and to obtain accurate temperature histories at the peak, and hence accurate drying temperatures, readings were continued for 5 hr.

The drying temperature, found from the autoclave temperature history depend on the peak temperature reached. The power to the autoclave was shut off 40 C before the desired temperature was reached. After the power was shut off, the temperature inside the autoclave will rise by about 40 C and then begin to decrease. The heating block temperature will decrease immediately after the power is shut off. The dry-

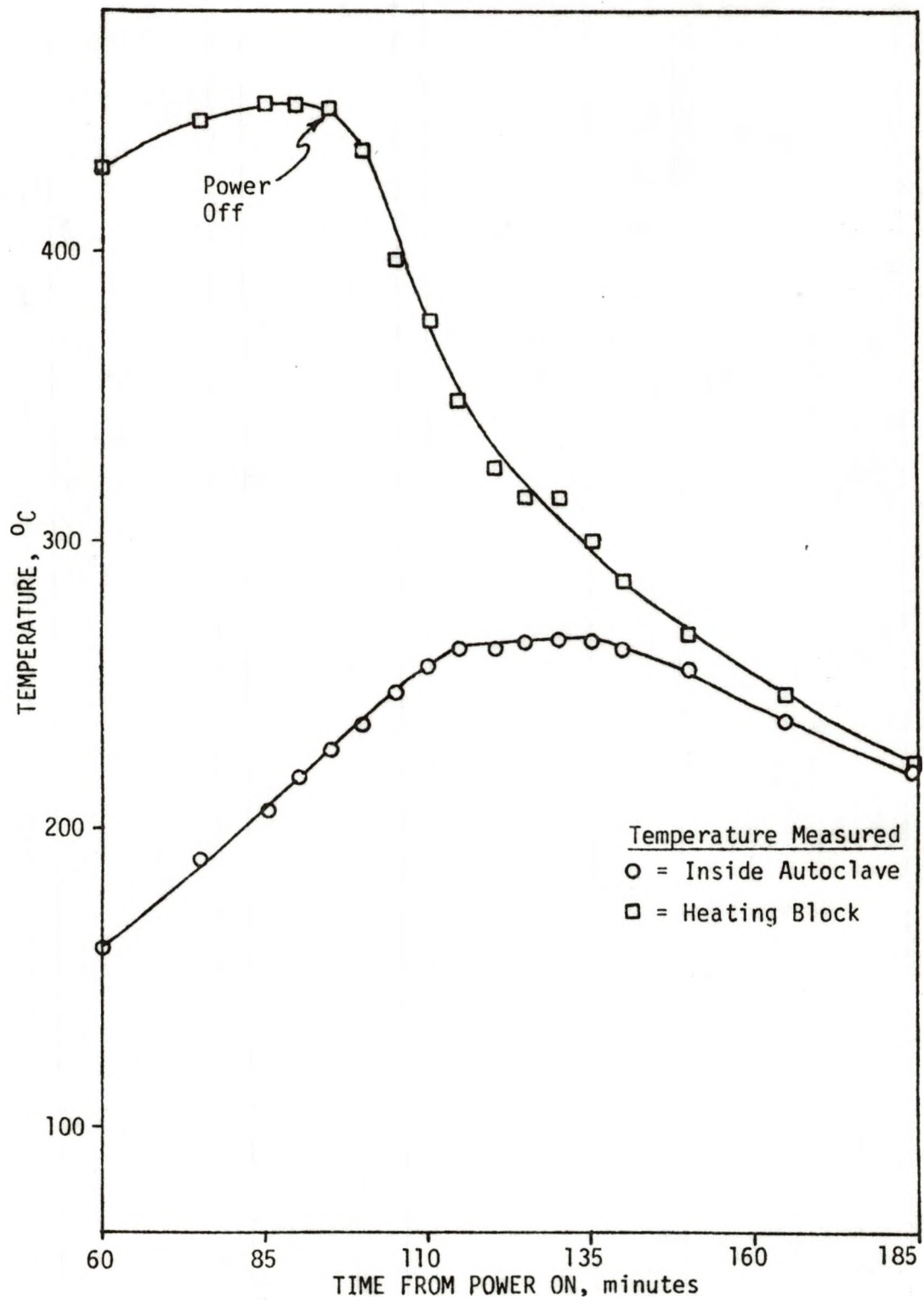


FIGURE 3 : Temperature history of autoclave interior and heating block.



ing temperature is defined as the temperature 2.5 C below the peak temperature reached for the autoclave interior (which is the same as that reached for the slurry), and the residence time is defined as the duration of time the slurry is within  $\pm 2.5$  C of the drying temperature. The residence time was kept constant at  $15 \pm 5$  min for all the runs by adjusting the insulation around the autoclave body. Figure 3 shows the temperature histories inside the autoclave and of the heating block.

#### REMOVAL OF PRODUCT FROM AUTOCLAVE

The autoclave and its contents were allowed to cool to room temperature for about 12 hrs. The residual gas in the autoclave was collected in a gas sampling bag for analysis using the gas sampling valve. The external piping system was detached from the autoclave and the autoclave cover was opened. The steel bucket containing the slurry was slowly pulled out of the autoclave with the help of hooked rod. There was water collected in the bottom of the autoclave beneath the bucket. This is the water that had condensed in the autoclave and collected in the space between the bucket and the autoclave walls. The bottom water was collected and combined with the slurry in the steel bucket with a pipette.

The hot water dried coal-water slurry was filtered for about 5-10 mins, using a Buchner funnel and a vacuum pump.

The filtrate was collected, weighed and stored under refrigeration for analysis. The hot water dried coal on the filter paper was also collected for analysis.

#### REMOVAL OF EXCESS MOISTURE FROM HOT WATER DRIED COAL

The hot water dried coal collected on the filter has excess moisture sticking onto its surface. It was spread onto 3-4 sheets (one on top of another) of light duty Kimwipes. With the help of another Kimwipe sheet the top of the coal layer was patted till all the Kimwipes were wet. The coal was then transferred to a new set of 3-4 sheets of Kimwipes, and the whole process was repeated till the Kimwipes absorbed no more moisture. The hot water dried coal was then assumed to be free of excess moisture. It still had surface moisture present on its surface. The surface moisture is actually the amount of moisture the coal took in when it was wetted with water.

Surface moisture is defined as the free moisture present in the top layers of the coal particle. Surface moisture does not include the excess moisture sticking to the surface of the coal particle. Hence, before determining the moisture content of the wetted coal, the excess moisture sticking to the surface of the particle is wiped off. The coal was weighed and stored in a glass bottle for analysis.



DETERMINATION OF SURFACE MOISTURE UPON WETTING

The following method was followed to determine the surface moisture of the original coal. The hot water dried coal after filtration and removal of excess moisture, contained inherent moisture and surface moisture. It was not possible to directly obtain quantitatively the amount of surface moisture present on the hot water dried coal. The amount of surface moisture present on the hot water dried coal was estimated by determining the amount of surface moisture on the original coal after it was wetted and the excess moisture wiped off, and assuming that the hot water dried coal would contain the same percentage of surface moisture. This was necessary since the inherent moisture in the original coal can be determined easily, but the same was not true for the hot water dried coal.

Approximately 15 g of the sieved coal of the given particle size was used for the purpose. Three samples of about 5 g each were placed in 3 different beakers, each with about 50 ml distilled water. The resulting mixture was stirred gently for 1/2 hr, 2 hrs, and 24 hrs respectively. The mixtures were then filtered for 5-10 mins using a Buchner funnel and a vacuum pump. The filtered coal was then wiped off the excess moisture sticking onto its surface using light duty Kimwipes as explained in the previous section.



The percent moisture in the original coal and the wetted original coal was determined. The percent moisture of the 3 batches of wetted coal were found to be similar. Knowing the amount of moisture in the original coal and wetted coal which was wiped, the surface moisture upon wetting could be determined. The details of the procedure and calculation of the amount of surface moisture upon wetting is shown in the Appendix B. A graph showing the relationship between the surface moisture upon wetting and particle size is also presented (Figure 20 in Appendix B). This graph shows the amount of surface moisture present on original coal particles of a certain mean particle diameter.

#### MOISTURE REABSORPTION TESTS USING A 100% HUMIDITY CHAMBER

An airtight glass desiccator which contained about 500 ml of distilled water was used as a humidity chamber. The coal samples were placed in the chamber and the water maintained the humidity inside the chamber at 100 %. The 100 % humidity chamber was used to determine the moisture reabsorption of the hot water dried and original coals. This was necessary in order to determine the effect of the drying temperature on the equilibrium moisture content of the hot water dried coal.

About 5 g of coal was accurately weighed into a previously weighed watch glass, and it was then put inside the 100%

humidity chamber. The watch glass and its contents were weighed every 24 hrs till they attained a constant weight (with slight variations) for 5 to 6 consecutive days. The coal was then assumed to have attained its equilibrium moisture content. The percent moisture of the coal was then determined. Knowing the equilibrium moisture content, the percent moisture of the coal could then be determined as a function of time the coal was in the chamber. Each day the watch glass and its contents were weighed. The details of the calculation of moisture content and equilibrium moisture content are shown in Appendix C.

## ANALYSES

### Analysis of coal

Proximate and ultimate analyses were performed on a sample of the original or as recieved coal used for each run as well as for the hot water dried coal produced. The analysis on each sample of original coal used is necessary since coal is heterogenous nature and its analysis varies from sample to sample. The ash was analyzed for sodium, potassium, magnesium, and calcium content. The calorific value and the carboxylic acid group content were also determined. Total sulfur present in the coal was determined by a Leco 532 automatic titrator. The details of calculation of moisture, ash, metals, and sulfur contents, and percent reductions of these are shown in the Appendices. Similar tests were also



performed on each sample of hot water dried coal. Percent ash and moisture were determined by the methods as specified by the American Society for Testing and Materials (ASTM) procedures #D3174 and # D3173 respectively (32).

The volatile matter and fixed carbon in the original coal were determined by ASTM procedure # D3175 (32). Nitrogen and sulfur contents were found using ASTM procedures # D3179 and # D1552 respectively (32), while carbon and hydrogen were determined using ASTM procedure # D3178 (32).

A Parr adiabatic oxygen bomb calorimeter was used to determine the calorific value of the coal (33). About 1.0 g of the original coal was burned in the calorimeter to determine the calorific value. The calorific value was corrected for the formation of nitric acid and sulphuric acids.

Carboxylic acid group content of the coal was determined by the following method (34). One hundred ml of about 0.2 N barium hydroxide were mixed well with 4 to 5 g of well powdered coal. The mixture was stirred continuously in a conical flask for 24 hrs. After 24 hrs of continuous stirring, the mixture was filtered, using a Buchner funnel and a vacuum pump. The filtered coal cake was then rinsed with distilled water, until it tested neutral using red litmus paper. The conical flask was rinsed three times with distilled water. Both rinses were collected and mixed with the filtrate. The filtrate was titrated potentiometrically



against a standard hydrochloric acid solution, using a Fisher Accumet model 230A pH/ion meter. The details of the calculation of the carboxylic acid group content of the coal are shown in Appendix J.

The ash of the coal was analyzed for sodium, potassium, magnesium, and calcium content (35). The following procedure was adopted for the determination of the metal contents of the coal. About 0.1 g of ash was accurately weighed and mixed with 6 times its weight of lithium metaborate. This mixture was thoroughly mixed in a plastic vial, transferred to a graphite crucible and heated to 975 C. During the heating period, the mixture was transformed from a powdery form to a shiny pellet. After maintaining the temperature (975 C) for 15 mins, the pellet was cooled to room temperature and dissolved in concentrated nitric acid. The resulting solution was diluted to 100 ml with distilled water. This solution was analyzed for sodium, potassium, magnesium, and calcium using a Perkin-Elmer model 303 atomic absorption spectrophotometer.

#### Analysis of Filtrate

The filtrate collected from the autoclave after each run was analyzed for sodium, potassium, magnesium, calcium, and sulfur. A Perkin-Elmer model 303 atomic absorption spectrophotometer was used to determine the sodium, potassium, magnesium, and calcium content of the filtrate (35). A Leco 532

automatic titrator was used to determine total sulfur in the filtrate. Calculations for sulfur and metals contents of the filtrate are shown in Appendix F and H respectively.

#### Analysis of the Residual Gas

The residual gas collected from the autoclave was collected in a 2-liter gas sampling bag after the autoclave had cooled down to room temperature. The gas was analyzed using a Hewlett Packard dual-column gas chromatograph with Porapak Q and 5A Mole Sieve columns. The Porapak Q column was used to detect and analyze for carbon dioxide, ethane and propane gases, and the Mole Sieve column was used to detect and analyze for hydrogen, oxygen, nitrogen, methane, and carbon dioxide gases. Ammonia content in the gas sample was determined by using the Nessler Method as found in APHA Standard Methods (36) while hydrogen sulfide was determined by ASTM Method # D2385 (32). Calculation of the carbon dioxide content of the residual gas is shown in Appendix K.

## Chapter V

### EXPERIMENTAL RUNS

Seventeen experimental runs to study the effect of temperature and particle size on hot water drying of the coal under investigation. Eight runs were made with mean particle diameter of 0.4699 cm and 9 runs were made with particles of 0.0505cm mean particle diameter. Table 2 lists the various runs made, along with the drying temperatures, particle size and the actual residence time at the various drying temperatures. Runs 1 to 3 were rejected due to leaks and malfunction of the instruments. The residence time was kept constant at  $15 \pm 5$  minutes.

The original coal was divided into 8 portions or groups. The as recieved coal in each portion was analysed. The coal from each portion was used for the several runs. The following list below shows the grouping of the runs made with each portion of the as recieved coal. The analysis for the original coal groups is shown in Table 27 and 28 in Appendix M.



TABLE 2

Details of the Experimental Runs

Run No.	Mean Particle Dia., cm	Drying Temperature, deg. C	Actual Residence Time, min
4	0.4699	282.5	16.50
5	0.4699	296	15.00
6	0.4699	335	13.00
7	0.4699	305	16.00
8	0.4699	311	18.00
9	0.4699	262.5	18.00
10	0.4699	327	16.50
11	0.4699	360	16.50
12	0.0505	325	18.00
13	0.0505	340	19.50
14	0.0505	313	18.00
15	0.0505	348	19.50
16	0.0505	298	19.50
17	0.0505	313	18.00
18	0.0505	328	18.00
19	0.0505	303	19.50
20	0.0505	343	16.50

TABLE 3

## Grouping of Coal for the Various Runs

Group Number	Grouping of Runs
1	Run #4
2	Runs #5, #6
3	Runs #7, #8
4	Run #9
5	Runs #10, #11
6	Run #12, #13, #14
7	Runs #15, #16, #17
8	Runs #18, #19, #20

## Chapter VI

### RESULTS AND DISCUSSION

The hot water drying runs were studied over a temperature range from 260 to 360 C. The residence time of the coal-water slurry at the drying temperatures was kept constant at  $15 \pm 5$  mins and the mass ratio of the solid to liquid in the slurry was held at 1:1 for all runs.

#### EFFECT OF DRYING TEMPERATURE ON MOISTURE REDUCTION

The moisture content of the as received coal or original coal (CAR), the hot water dried (HWD) coal, and the percent moisture reduction upon drying are tabulated in Table 4 for the two particle sizes studied. Figure 4 is a plot of the moisture reduction as a function of the drying temperature for the 0.4699 cm and 0.0505 cm mean diameter particles. Appendix A show a sample calculation for the moisture content in coal.

The moisture reduction was found to be independent of the drying temperature for both the particle sizes. The reduction in moisture was found to be independent of the particle size for the temperature range investigated. The average moisture reduction for 0.4699 cm mean particle diameter was



72.5 percent and was 77.3 percent for the 0.0505 cm mean particle diameter. This is probably due to the fact that smaller particles have a shorter distance for the moisture to be transported through the pores of the coal compared to the larger particles and hence it results in a greater moisture removal. Maas (29) has shown for the hot water drying of a North Dakota lignite using cold charge autoclave that moisture removal was a linear function of drying temperature in the range between 200 to 285 C.

A statistical analysis (t-test) was done, at a 5 percent significance level, on the moisture reductions for the two particle sizes to determine whether there is any significant difference between the moisture reductions for the 2 particle sizes. From the analysis it was concluded that there is significantly no difference between the moisture reductions for the two particle sizes. Please refer to Appendix N for the details of the statistical analysis.

It was observed by Baria, Mass, and Paulson (3) that at drying temperatures higher than 320 C for hot water drying of a North Dakota lignite in hot charge autoclave, the moisture removal was independent of temperature. These observations suggests that at lower temperatures moisture removal is a function is a function of temperature and at higher temperatures moisture removal is independent of temperature since most of the water has already been removed at the low-

TABLE 4

Moisture Reduction and Inherent Moisture Contents of  
Original Coal and Hot Water Dried Coal

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Inherent Moisture Content, %		Moisture Reduction, %
			CAR	HWD Coal	
4	0.4699	282.5	23.90	8.20	75.11
5	0.4699	296	25.18	9.81	72.64
6	0.4699	335	25.18	8.54	75.58
7	0.4699	305	23.60	9.93	70.43
8	0.4699	311	23.60	8.02	75.85
9	0.4699	262.5	27.80	9.91	73.06
10	0.4699	327	26.87	11.06	70.40
11	0.4699	360	26.87	12.62	67.13
12	0.0505	325	26.94	14.20	60.22
13	0.0505	340	26.94	12.35	66.51
14	0.0505	313	26.94	10.19	74.29
15	0.0505	348	28.84	9.80	76.90
16	0.0505	298	28.84	9.53	77.40
17	0.0505	313	28.84	7.47	82.32
18	0.0505	328	28.83	4.43	90.81
19	0.0505	303	28.83	6.37	85.31
20	0.0505	343	28.83	8.03	81.57

er temperatures. The temperature at which the moisture removal becomes independent of temperature is a function of the original moisture content of the coal, the type of coal studied, and particle size.

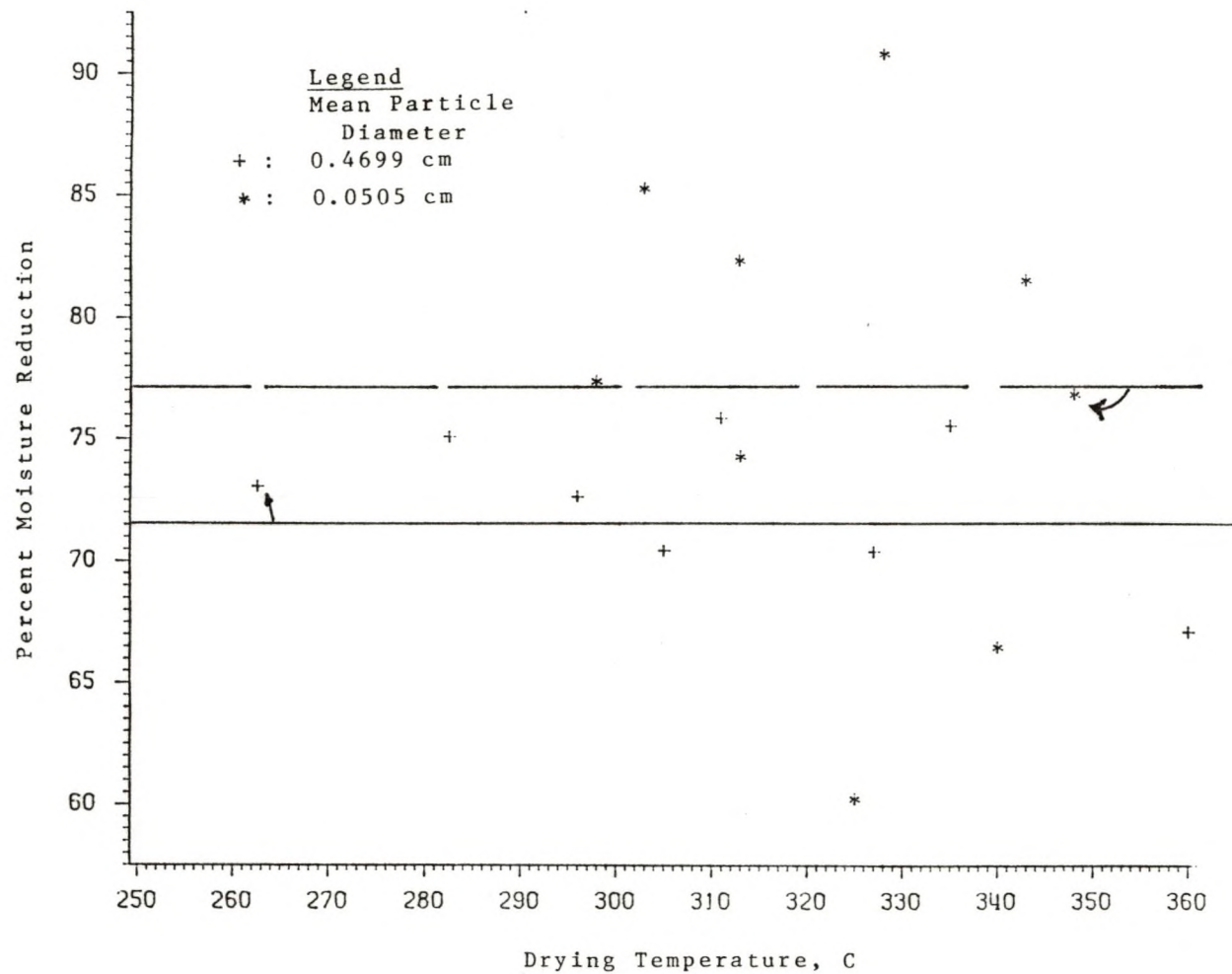


Figure 4: Moisture Reduction as a Function of Drying Temperature



EFFECT OF DRYING TEMPERATURE ON MOISTURE REABSORPTION AND  
THE EQUILIBRIUM MOISTURE CONTENT

The moisture content of the original coal and the hot water dried coal as a function of the number of days in the humidity chamber is tabulated in Table 5 for 0.4699 cm mean particle diameter coal and in Table 6 for 0.0505 cm mean particle diameter coal. The values used for the original coals are for the coal used for Run #4 for 0.4699 cm particle diameter, and Runs #17 for 0.0505 cm particle diameter. The moisture content of the various other original coals for both particle sizes as a function of time is listed in Table 26 in the Appendix D. Figure 5 and 7 are the plots of moisture content of original coals used for run #5 (0.4669 cm mean particle diameter) and run #18 (0.0505 cm mean particle diameter) respectively. Figures 6 and 8 are the plots of moisture content of hot water dried coal as a function of time in humidity chamber for varying drying temperatures for 0.4699 cm and 0.0505 cm particle diameter respectively. Similar graphs for the original coal are plotted in Figure 21 and 22 in Appendix D. Appendix D also shows the method of determination of the equilibrium moisture content.

The moisture content of the hot water dried coal and original coal attained a constant value after 5 days. This constant moisture content is defined as the equilibrium moisture content of the coal. It was observed that the equilibrium moisture content of the hot water dried coals

TABLE 5

Moisture Reabsorption Tests on 0.4699 cm Mean Particle  
Diameter HWD Coal in 100 % Humidity Chamber

Time, days	Percent Moisture in Coal								
	Original	Hot Water Dried Coal at							
	Coal	282 C	296 C	335 C	305 C	311 C	262 C	327 C	360 C
	Run#7	Run#4	Run#5	Run#6	Run#7	Run#8	Run#9	Run#10	Run#11
0	22.65	13.96	13.46	12.22	15.58	14.52	11.56	13.00	12.00
1	22.10	12.41	12.10	11.18	12.88	10.99	12.21	11.64	11.01
2	21.77	12.38	12.04	11.48	11.77	10.80	12.41	11.27	10.70
3	21.45	12.37	11.98	11.65	11.56	10.52	12.62	10.37	09.38
4	21.66	12.36	12.04	11.16	11.36	09.77	12.29	09.81	09.04
5	21.71	12.49	12.12	11.22	11.41	09.93	12.58	09.80	08.87
6	21.74	12.74	12.28	11.35	11.46	10.25	12.86	09.78	08.90
7	21.77	12.76	12.34	11.38	11.49	10.37	12.86	09.80	08.90
8	21.75	12.82	12.45	11.42	11.53	10.37	12.95	09.80	08.90
9	21.77	12.78	12.34	11.27	11.44	10.29	12.89	09.98	08.90
10	21.80	12.78	12.34	11.35	11.51	10.34	12.91	09.80	08.90
11	21.83	12.84	12.43	11.42	11.60	10.37	12.96	09.80	08.90
12	21.86	12.92	12.53	11.50	11.67	10.42	13.02	09.80	08.90
13	21.66	12.97	12.61	11.60	11.75	10.47	13.07	09.80	08.90
14	21.38	12.97	12.57	11.33	11.55	10.34	13.05	09.80	08.90
16	21.31	12.75	12.44	11.52	11.45	10.32	12.88	09.80	08.90



TABLE 6

Moisture Reabsorption Tests on 0.0505 cm Mean Particle  
Diameter HWD Coal in 100 % Humidity Chamber

Time, days	Percent Moisture in Coal									
	-----									
	Original	Hot Water Dried Coal at								
	Coal	325 C	340 C	313 C	348 C	298 C	313 C	328 C	303 C	343 C
	Run#17	Run#12	Run#13	Run#14	Run#15	Run#16	Run#17	Run#18	Run#19	Run#20
0	21.00	19.80	19.30	20.30	18.00	21.20	20.10	19.80	20.50	18.90
1	21.75	18.95	18.03	18.60	16.31	19.51	18.74	16.98	19.09	17.15
2	22.05	17.67	17.16	17.77	15.61	17.88	17.21	16.26	17.96	16.20
3	22.52	16.73	16.45	17.01	14.44	17.59	16.91	15.78	17.31	15.11
4	22.82	16.17	15.75	16.80	14.21	17.19	16.52	15.62	16.81	14.98
5	22.95	15.90	15.55	16.65	14.08	17.15	16.40	15.56	16.50	14.78
6	23.05	15.87	15.20	16.38	14.00	17.10	16.34	15.60	16.50	14.59
7	23.05	15.88	15.16	16.10	14.00	17.10	16.30	15.60	16.50	14.50
8	23.05	15.87	15.12	16.10	14.00	17.10	16.30	15.65	16.60	14.50
9	23.05	15.90	15.10	16.08	14.00	17.05	16.30	15.60	16.54	14.50
10	23.05	15.90	15.10	16.05	14.00	17.05	16.30	15.60	16.55	14.50
11	23.05	15.90	15.10	16.10	14.00	17.10	16.30	15.60	16.60	14.50
12	23.05	15.90	15.10	16.10	14.00	17.10	16.30	15.60	16.60	14.50
13	23.05	15.87	15.10	16.10	14.00	17.10	16.30	15.60	16.60	14.50
14	23.05	15.87	15.10	16.10	14.00	17.10	16.30	15.60	16.60	14.50
15	23.05	15.90	15.10	16.10	14.00	17.10	16.30	15.60	16.60	14.50
16	23.05	15.90	15.10	16.10	14.00	17.10	16.30	15.60	16.60	14.50



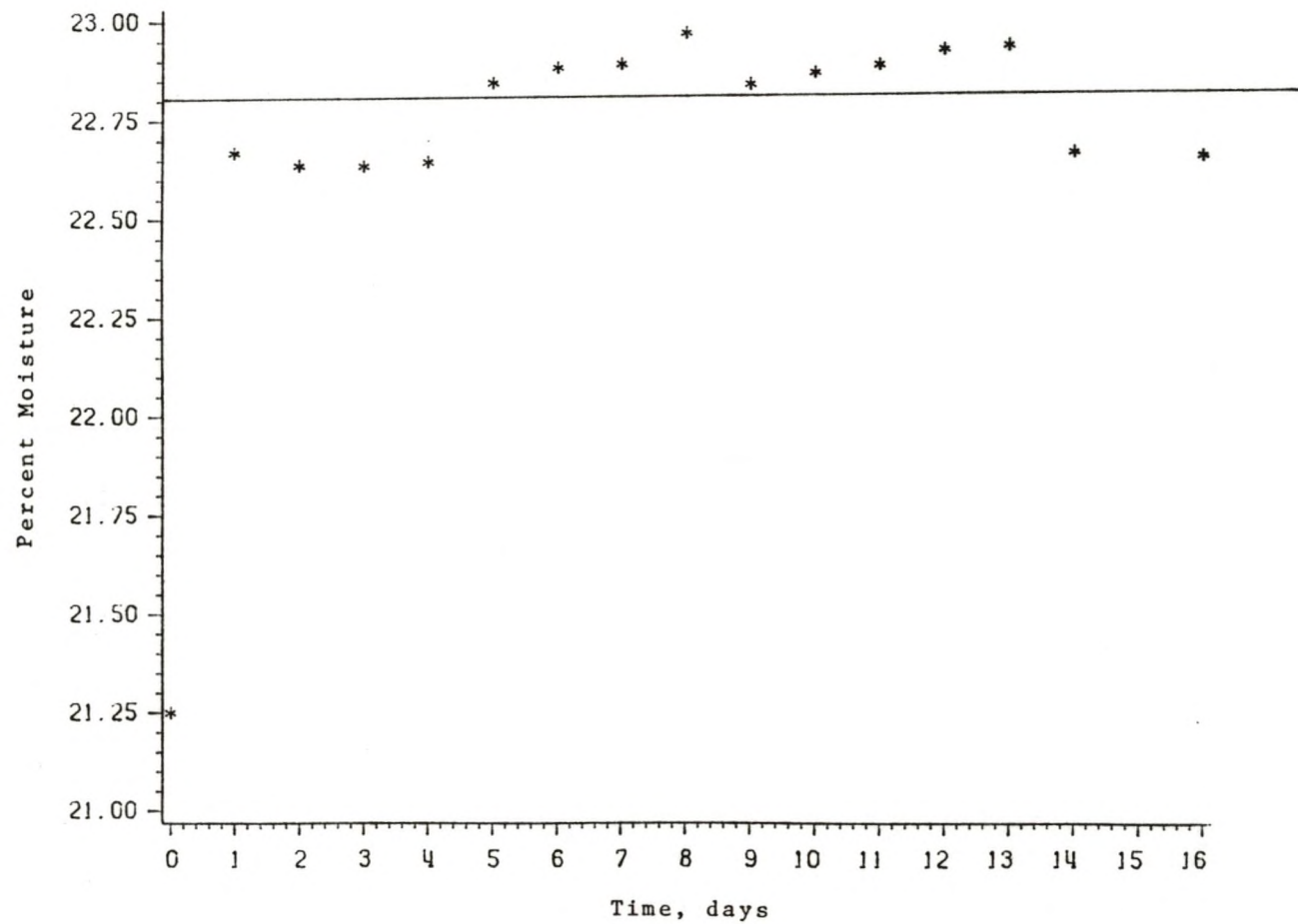


Figure 5: Moisture Reabsorption by Original Coal (0.4699 cm Mean Diameter Particles) used for Run #5 in 100% Humidity Chamber

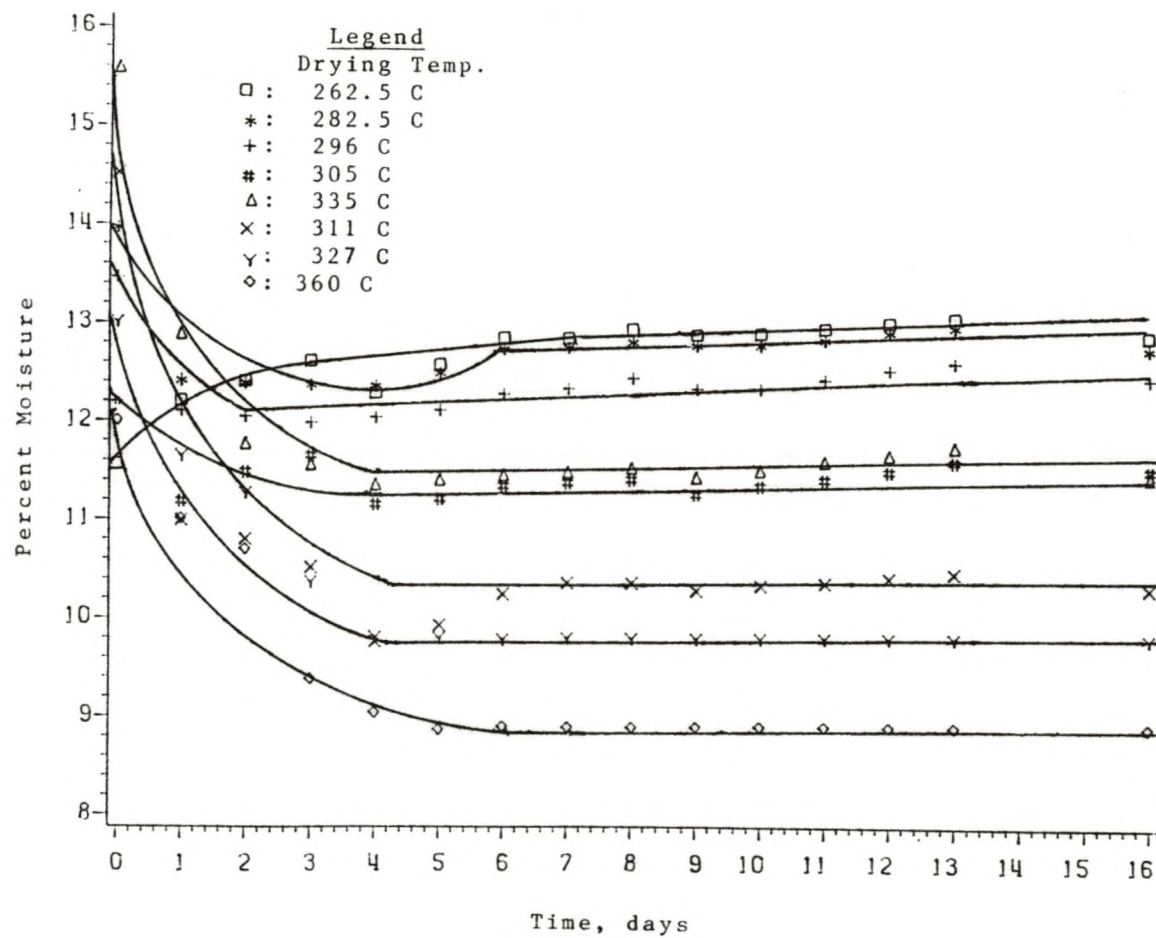


Figure 6: Moisture Reabsorption by Hot Water Dried Coal (0.4699 cm mean diameter particles) in 100 % Humidity Chamber

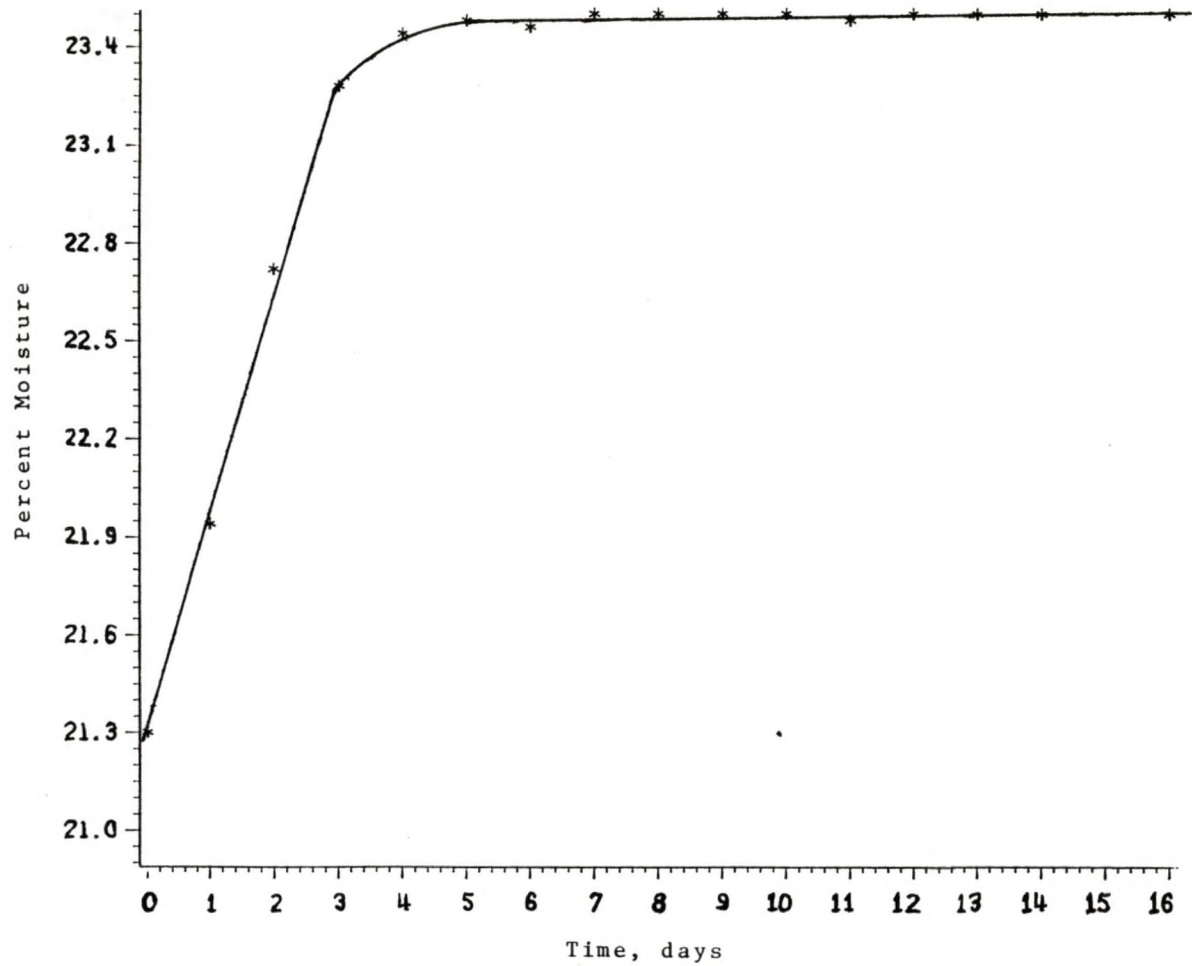


Figure 7: Moisture Reabsorption by Original Coal (0.0505 cm Mean Diameter Particles) used for Run #18 in 100% Humidity Chamber



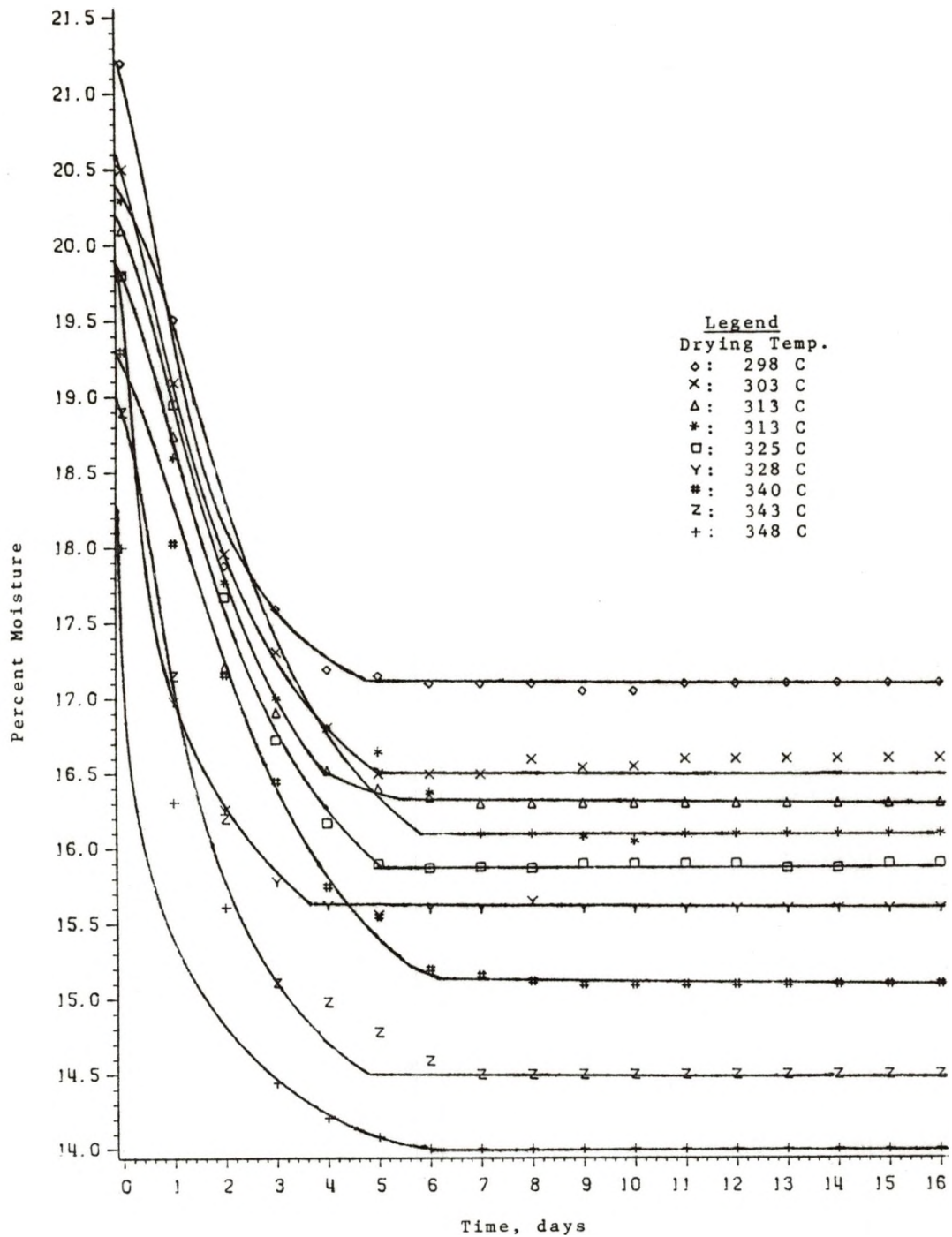


Figure 8: Moisture Reabsorption by Hot Water Dried Coal (0.0505 cm mean diameter particles) in 100% Humidity Chamber

decreased as the drying temperature increased. This is better seen in Table 7 which tabulates the equilibrium moisture contents of the original and hot water dried coals for the various drying temperatures. Figure 9 is a plot of equilibrium moisture content as a function of the drying temperature for the two particle sizes. It was observed that the equilibrium moisture content for the larger particles is less than that of the smaller particles. This is due to increased surface area per unit mass of the coal for the smaller particles, and hence increased amount of water it can hold per unit mass.

Mass (29) also observed a decrease in equilibrium moisture content with increase in drying temperature. The decrease in equilibrium moisture content with increase in drying temperature is probably due to the destruction of the hydrophillic carboxylic groups, due to evolution of CO<sub>2</sub>, and replacement by hydrophobic groups, as the drying temperature increases.

TABLE 7

Equilibrium Moisture Content of HWD Coal and CAR

Run No.	Mean Particle Dia., cm	Drying Temperature, deg. C	Equilibrium Moisture Content, %	
			HWD Coal	CAR
4	0.4699	282.5	12.60	21.10
5	0.4699	296	12.50	23.00
6	0.4699	335	11.50	23.00
7	0.4699	305	11.60	21.70
8	0.4699	311	10.30	21.70
9	0.4699	262.5	13.00	22.80
10	0.4699	327	09.60	22.00
11	0.4699	360	08.80	22.00
12	0.0505	325	15.60	24.00
13	0.0505	340	15.00	24.00
14	0.0505	313	16.10	24.00
15	0.0505	348	13.80	23.10
16	0.0505	298	17.00	23.10
17	0.0505	313	16.20	23.10
18	0.0505	328	15.40	23.50
19	0.0505	303	16.40	23.50
20	0.0505	343	14.40	23.50



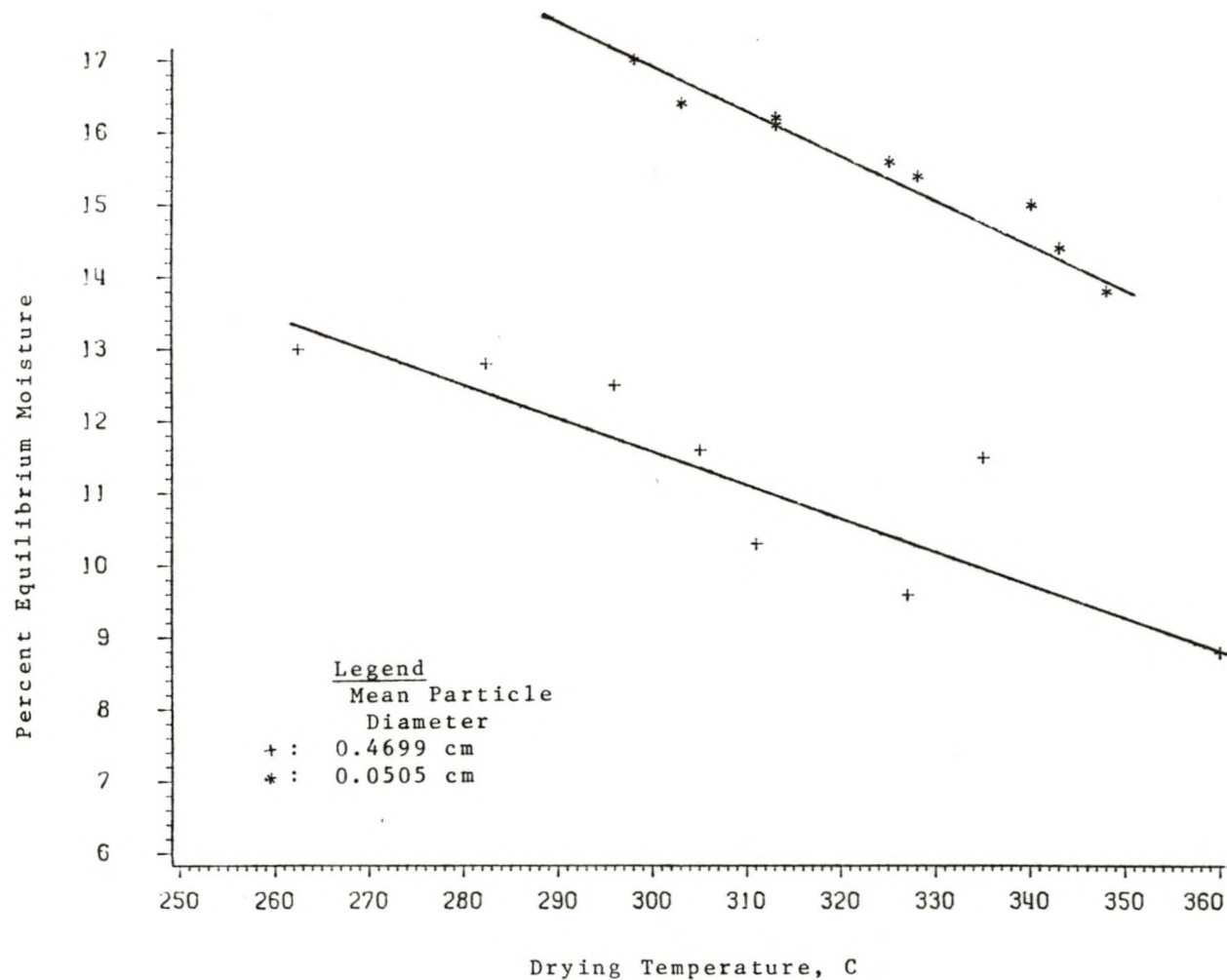


Figure 9: Equilibrium Moisture Content of Hot Water Dried Coal  
as a Function of Drying Temperature

EFFECT OF DRYING TEMPERATURE ON ASH CONTENT OF COAL

The ash content of the original and hot water dried coals are tabulated in Table 8 for the two particle sizes studied. Figure 10 is a plot of the ash content of the hot water dried coal as a function of the drying temperature for the two particle sizes. It was observed that there was no significant change in the ash content of the coal upon hot water drying. This is consistent with the fact that there should be no change in the ash content since most of the coal molecule is not affected by this drying process. Appendix A shows a sample calculation for the ash content in coal.

A statistical analysis (t-test) was performed at a 5 percent significance level on the ash content of the hot water dried coal to determine if there is any significant difference between the ash contents of the hot water dried coal and the original coal (CAR). From the statistical analysis it was determined that the ash contents of the hot water dried coal and the original coal was significantly not different. Please refer to Appendix N for the details of the statistical analysis.

TABLE 8

Ash Content of CAR and HWD Coal

Run No.	Mean Particle Dia., cm	Drying Temperature, deg. C	Ash Content(dry basis), %	
			CAR	HWD Coal
4	0.4699	282.5	10.20	15.30
5	0.4699	296	13.14	11.24
6	0.4699	335	13.14	12.49
7	0.4699	305	11.90	13.50
8	0.4699	311	11.90	12.60
9	0.4699	262.5	12.43	12.69
10	0.4699	327	13.11	14.90
11	0.4699	360	13.11	13.93
12	0.0505	325	18.60	19.65
13	0.0505	340	18.60	18.61
14	0.0505	313	18.60	19.23
15	0.0505	348	21.59	21.73
16	0.0505	298	21.59	20.81
17	0.0505	313	21.59	22.44
18	0.0505	328	21.54	21.76
19	0.0505	303	21.54	20.82
20	0.0505	343	21.54	22.45



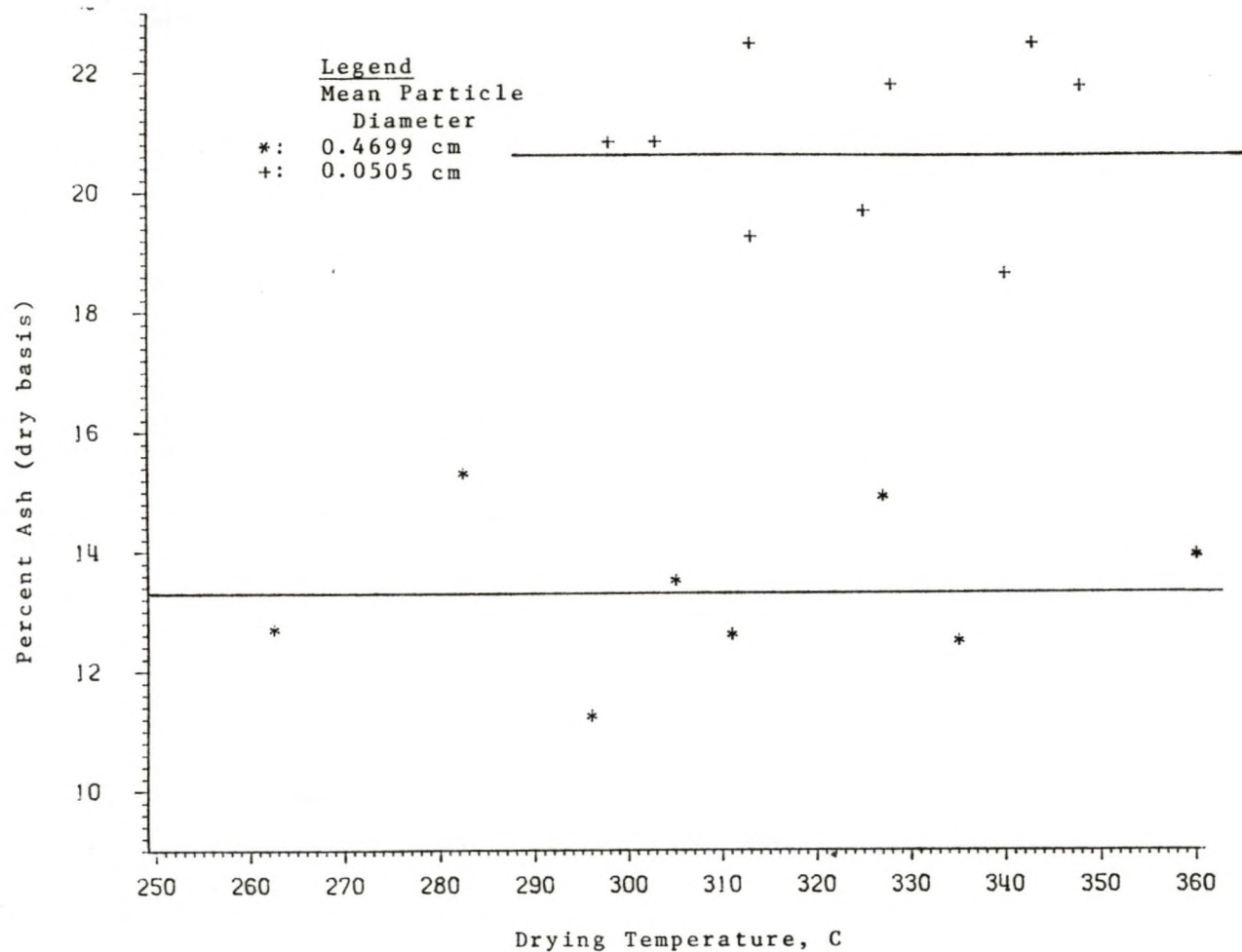


Figure 10: Ash Content (dry basis) of Hot Water Dried Coal as a Function of Drying Temperature

EFFECT OF DRYING TEMPERATURE ON SULFUR REMOVAL

The distribution of sulfur in the original coal upon hot water drying was examined. As discussed in a later section no or negligible sulfur was present in the residual gas as hydrogen sulfide or sulfur dioxide. Sulfur was found to be present in the filtrate and in the dried coal. A material balance was performed to determine the distribution of the sulfur in the as recieved coal between the filtrate and the dried coal. Table 9 lists the amount of sulfur present in the original coal, hot water dried coal, and the filtrate per 150 g of as recieved coal processed for each drying temperature and the material balance closure obtained. It is observed that the material balance closure varies from 51 to 165 percent.

The sulfur content of the original coal and the hot water dried coal and the extent of sulfur removal (normalised) are listed for both particle sizes for the various drying temperatures in Table 10. The percent sulfur removal (normalised) is plotted as a function of the drying temperature in Figure 11. Sulfur removal on hot water drying was found to be low. The sulfur removal varies from 0 to 15 percent and sulfur removal decreases with increase in drying temperature for both the drying temperature. Statistical analysis was done with a 5 percent significance level. From the statistical analysis (t-test) it was concluded that the sulfur re-

moval was significantly different from 0 percent for both the particle sizes, that is, sulfur was removed. The average sulfur removal was low for both the particle sizes. For the statistical analysis please refer to Appendix N.

TABLE 9  
Material Balance Closures for Sulfur

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Sulfur Content, g/150 g CAR	HWDCoal	Filtrate	Material Balance Closure, %
4	0.4699	282.5	0.589	0.503	0.087	128
5	0.4699	296	0.518	0.440	0.078	87
6	0.4699	4335	0.518	0.481	0.038	99
7	0.4699	305	0.531	0.482	0.055	112
8	0.4699	311	0.531	0.530	0.000	94
9	0.4699	262.5	0.703	0.650	0.053	100
10	0.4699	327	0.450	0.416	0.033	165
11	0.4699	360	0.450	0.401	0.049	100
12	0.0505	325	1.15	1.08	0.030	75
13	0.0505	340	1.15	1.17	0.038	78
14	0.0505	313	1.15	1.04	0.530	70
15	0.0505	348	1.29	1.27	0.093	54
16	0.0505	298	1.29	1.29	0.118	51
17	0.0505	313	1.29	1.28	0.084	77
18	0.0505	328	1.04	1.00	0.053	85
19	0.0505	303	1.04	0.998	0.082	79
20	0.0505	343	1.04	0.927	0.050	99

Previous work by Maas (29), Elliot (20) and Koppelman (12) have indicated removal of 10 percent to 20 percent of the sulfur present in the coal on hot water drying. This is consistent with the sulfur removal observed in this study. The extent of sulfur removal is a function of the type of



TABLE 10

## Sulfur Removal on Hot Water Drying

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Sulfur Content (dry basis), %		Sulfur Removed (normalised), %
			CAR	HWD Coal	
4	0.4699	282.5	0.516	0.647	14.80
5	0.4699	296	0.473	0.353	15.20
6	0.4699	335	0.473	0.491	7.40
7	0.4699	305	0.470	0.490	10.40
8	0.4699	311	0.470	0.450	0.00
9	0.4699	262.5	0.650	0.630	7.52
10	0.4699	327	0.424	0.742	7.33
11	0.4699	360	0.424	0.451	10.90
12	0.0505	325	1.09	0.902	2.79
13	0.0505	340	1.09	0.936	3.08
14	0.0505	313	1.09	0.863	5.57
15	0.0505	348	1.37	0.855	6.97
16	0.0505	298	1.37	0.741	8.23
17	0.0505	313	1.37	1.09	6.49
18	0.0505	328	1.04	1.05	4.94
19	0.0505	303	1.04	0.870	7.57
20	0.0505	343	1.04	1.13	5.18

sulfur present in the coal. The type of sulfur present in the coal under study was not investigated and hence no conclusions can be made to this effect. Appendices F and G show sample calculations for the sulfur content and the sulfur removal on hot water drying respectively.

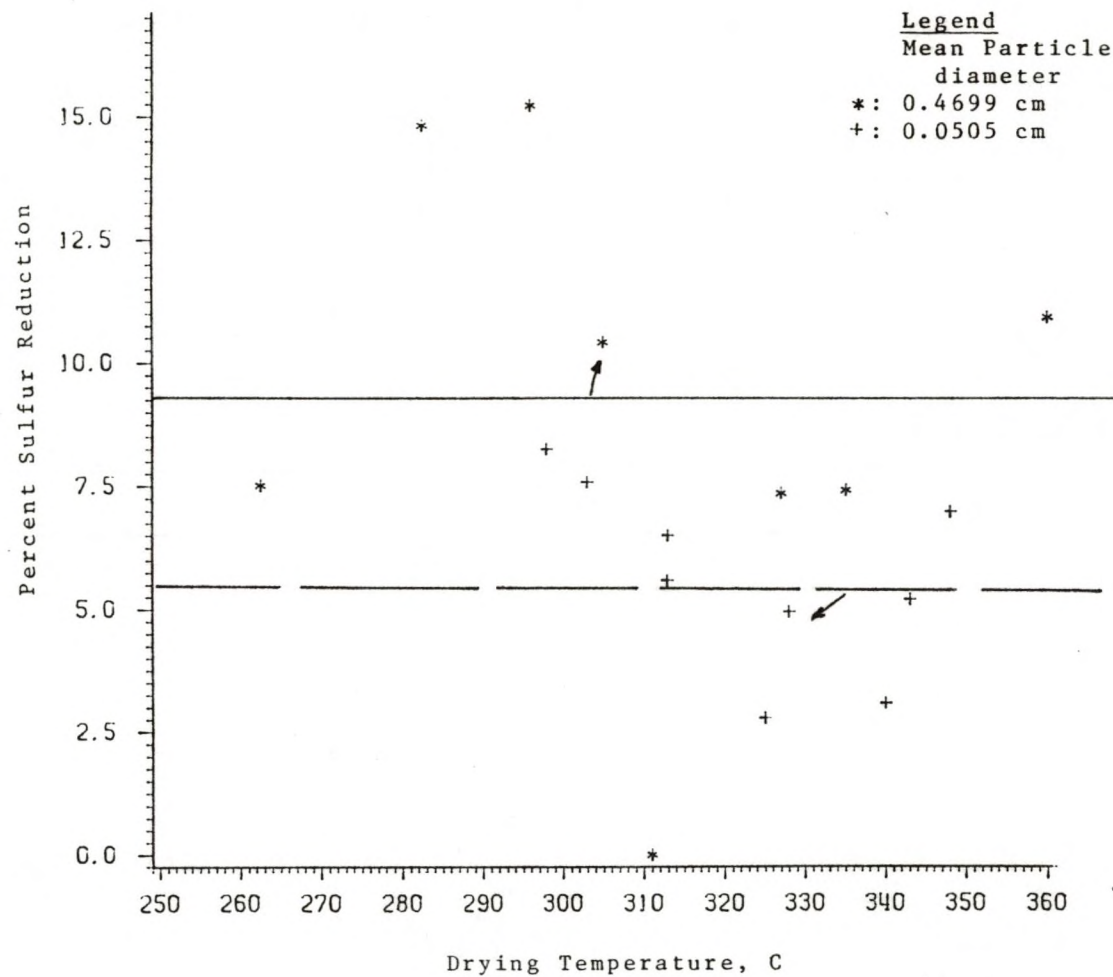


Figure 11: Sulfur Reduction (normalized) as a Function of Drying Temperature

EFFECT OF DRYING TEMPERATURE ON REMOVAL OF INORGANIC METALS

The inorganic metals, present in the ash, studied in this investigation were sodium, potassium, magnesium and calcium. These metals are distributed between the dried coal and filtrate upon hot water drying. Tables 11 to 14 show the amount of metal present in the original and hot water dried coals and the filtrate per 150 g of as received coal used and the material balance closure for various drying temperatures for sodium, potassium, magnesium and calcium respectively. The material balance closures vary from 59% to 147% for sodium, from 171% to 67% for potassium, from 181% to 71% for Magnesium and 108% to 57% for Calcium. These closures are in the acceptable range except for potassium. The wide range for potassium is probably due to the very low potassium content of the coal which is within the experimental error of the atomic absorption spectrophotometer and hence causes a large scatter in the data. Since the more common way of showing the composition of inorganic metals is in the form of percent oxide in ash, the composition of the metals in the coal based on metal oxide in ash is tabulated in Table 15 for various drying temperatures for both original and hot water dried coals.

The metal content in the original and hot water dried coals and the percent removed (normalised) are tabulated in Tables 16 to 19 for sodium, potassium, magnesium and calcium



TABLE 11

## Material Balance Closure for Sodium

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Sodium Content, g/150 g CAR			Material Balance Closure, %
			CAR	HWD Coal	Filtrate	
4	0.4699	282.5	0.236	0.110	0.127	115
5	0.4699	296	0.266	0.120	0.145	82
6	0.4699	335	0.266	0.131	0.136	59
7	0.4699	305	0.298	0.107	0.192	75
8	0.4699	311	0.298	0.101	0.195	70
9	0.4699	262.5	0.218	0.088	0.130	111
10	0.4699	327	0.249	0.564	0.192	70
11	0.4699	360	0.249	0.068	0.181	71
12	0.0505	325	0.221	0.082	0.134	106
13	0.0505	340	0.221	0.087	0.150	100
14	0.0505	313	0.221	0.073	0.140	102
15	0.0505	348	0.206	0.065	0.136	77
16	0.0505	298	0.206	0.128	0.081	127
17	0.0505	313	0.206	0.085	0.123	91
18	0.0505	328	0.189	0.068	0.124	94
19	0.0505	303	0.189	0.098	0.098	147
20	0.0505	343	0.189	0.034	0.143	91

for various drying temperatures. Figures 12,13,14,15 are plots of percent metal removed versus drying temperature for sodium, potassium, magnesium and calcium. The percentage removal for each metal studied, except sodium, is observed to be independent of drying temperature and particle size. For sodium percent removed (normalised) increases as drying temperature increases. The average value for sodium removal is 61.8 percent, for potassium removal is 19.2 percent, for magnesium removal is 3.9 percent, and for calcium removal is 13.2 percent.

TABLE 12

## Material Balance Closure for Potassium

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Potassium Content, g/150 g CAR			Material Balance Closure, %
			CAR	HWD Coal	Filtrate	
4	0.4699	282.5	0.079	0.076	0.0036	167
5	0.4699	296	0.093	0.081	0.0127	67
6	0.4699	335	0.094	0.085	0.0099	78
7	0.4699	305	0.088	0.080	0.0071	90
8	0.4699	311	0.087	0.081	0.0059	70
9	0.4699	262.5	0.095	0.059	0.0359	171
10	0.4699	327	0.120	0.115	0.0049	89
11	0.4699	360	0.120	0.113	0.0073	81
12	0.0505	325	0.221	0.217	0.0054	106
13	0.0505	340	0.242	0.234	0.0065	87
14	0.0505	313	0.219	0.212	0.0073	86
15	0.0505	348	0.248	0.239	0.0087	69
16	0.0505	298	0.252	0.250	0.0072	82
17	0.0505	313	0.255	0.249	0.0065	90
18	0.0505	328	0.196	0.191	0.0048	115
19	0.0505	303	0.200	0.199	0.0008	107
20	0.0505	343	0.181	0.180	0.0015	95

Maas (29) studied the sodium removal for different slurry concentrations and obtained averages of percent sodium removal based on the solid residue to range from 38 to 48 %. Since the sodium content of coal is related to the amount of ash fouling of heat exchanger tubes in boilers, it is the most important metal studied. Maas (29) obtained average percentage reductions in calcium, magnesium, and potassium of 0, 5, and 9 respectively. These metal reductions were based on filtrate data. He noticed the reduction of each metal to increase with drying temperature. Maas (29) no-



TABLE 13

## Material Balance Closure for Magnesium

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Magnesium Content, g/150 g CAR			Material Balance Closure, %
			CAR	HWD Coal	Filtrate	
4	0.4699	282.5	0.185	0.176	0.009	91
5	0.4699	296	0.256	0.250	0.006	82
6	0.4699	335	0.258	0.252	0.006	74
7	0.4699	305	0.327	0.319	0.007	75
8	0.4699	311	0.324	0.320	0.004	71
9	0.4699	262.5	0.228	0.219	0.010	110
10	0.4699	327	0.243	0.235	0.008	97
11	0.4699	360	0.244	0.242	0.002	99
12	0.0505	325	0.302	0.302	0.001	123
13	0.0505	340	0.331	0.330	0.002	104
14	0.0505	313	0.300	0.300	0.009	181
15	0.0505	348	0.264	0.260	0.004	93
16	0.0505	298	0.273	0.269	0.004	74
17	0.0505	313	0.271	0.269	0.002	94
18	0.0505	328	0.288	0.285	0.002	99
19	0.0505	303	0.293	0.290	0.003	87
20	0.0505	343	0.266	0.264	0.002	83

ticed that the ease of removal of metals due to hot water drying lignite was in the decreasing order of sodium, potassium, magnesium, and calcium. Sodium and potassium are removed very easily due to their small size and univalent charge.

In the present study, of the four metals studied, sodium is the easiest to remove. The decomposition of the acid functional groups may be a factor in the metal removal since these metals are associated with the acid functional groups



TABLE 14

## Material Balance Closure for Calcium

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Calcium Content, g/150 g CAR			Material Balance Closure, %
			CAR	HWD Coal	Filtrate	
4	0.4699	282.5	1.63	1.52	0.110	77
5	0.4699	296	1.87	1.79	0.075	84
6	0.4699	335	1.88	1.77	0.108	72
7	0.4699	305	2.68	2.58	0.102	75
8	0.4699	311	2.66	2.56	0.099	77
9	0.4699	262.5	1.62	1.54	0.084	101
10	0.4699	327	1.83	1.73	0.097	107
11	0.4699	360	1.84	1.68	0.167	108
12	0.0505	325	1.92	1.79	0.128	108
13	0.0505	340	2.10	2.10	0.000	90
14	0.0505	313	1.90	1.84	0.065	70
15	0.0505	348	2.22	2.06	0.163	61
16	0.0505	298	2.30	2.12	0.184	57
17	0.0505	313	2.29	1.47	0.821	101
18	0.0505	328	1.93	1.78	0.151	67
19	0.0505	303	1.97	1.78	0.193	59
20	0.0505	343	1.79	1.60	0.189	61

in coals in which chlorine is not present. In Australian brown coals, sodium is present as sodium chloride, and appreciable amounts of sodium removal, leached out as sodium chloride, were observed by Murray and Evans (26). They observed that sodium removal increased with temperature. Appendices H and I show sample calculations for the metals content and the metals removal on hot water drying respectively.

TABLE 15

Metal Oxides Content in Ash of Original Coal and HWD Coal

Run No.	Mean Particle Dia., cm	Run Temp., deg. C	Metal Oxide Content in Ash, Percent							
			Original Coal				HWD Coal			
			Na2O	K2O	MgO	CaO	Na2O	K2O	MgO	CaO
4	0.4699	282.5	3.10	0.82	2.60	19.6	1.10	1.00	1.80	11.0
5	0.4699	296	2.40	0.75	2.80	17.5	1.20	0.62	3.30	20.4
6	0.4699	335	2.40	0.75	2.80	17.5	1.00	0.78	3.00	17.4
7	0.4699	305	3.00	0.78	4.04	27.9	0.87	0.69	3.16	21.6
8	0.4699	311	3.00	0.78	4.04	27.9	0.79	0.57	3.12	22.7
9	0.4699	262.5	2.20	0.88	2.80	16.9	1.00	0.09	3.10	16.8
10	0.4699	327	2.40	1.00	2.92	18.5	0.38	0.87	2.76	18.9
11	0.4699	360	2.4	1.00	2.92	18.5	0.52	0.86	3.23	20.5
12	0.0505	325	1.50	1.41	2.67	14.2	0.67	1.56	3.50	15.3
13	0.0505	340	1.50	1.41	2.67	14.2	0.64	1.36	3.15	14.5
14	0.0505	313	1.50	1.41	2.67	14.2	0.65	1.36	5.59	27.8
15	0.0505	348	1.30	1.40	2.10	14.6	0.40	1.13	2.30	10.0
16	0.0505	298	1.30	1.40	2.10	14.6	1.20	1.33	1.80	9.18
17	0.0505	313	1.30	1.40	2.10	14.6	0.50	1.34	2.10	10.3
18	0.0505	328	1.20	10.8	2.20	12.4	0.50	1.49	2.60	9.38
19	0.0505	303	1.20	10.8	2.20	12.4	1.00	1.36	2.20	7.76
20	0.0505	343	1.20	10.8	2.20	12.4	0.20	1.14	2.00	7.52

TABLE 16

## Sodium Removal on Hot Water Drying

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Sodium Content (per g MAF coal Basis), mg		Sodium Removed (normalised), %
			CAR	HWD Coal	
4	0.4699	282.5	2.61	1.46	53.80
5	0.4699	296	2.84	1.15	54.72
6	0.4699	335	2.84	0.905	50.90
7	0.4699	305	3.01	1.01	64.20
8	0.4699	311	3.01	0.848	65.90
9	0.4699	262.5	2.30	1.09	59.40
10	0.4699	327	2.72	0.497	77.30
11	0.4699	360	2.72	0.627	72.70
12	0.0505	325	2.58	1.22	62.30
13	0.0505	340	2.58	1.08	63.70
14	0.0505	313	2.58	1.14	65.70
15	0.0505	348	2.64	0.769	67.50
16	0.0505	298	2.64	2.32	38.70
17	0.0505	313	2.64	1.12	59.20
18	0.0505	328	2.40	1.10	64.60
19	0.0505	303	2.40	2.22	50.00
20	0.0505	343	2.40	0.497	80.70



TABLE 17

## Potassium Removal on Hot Water Drying

Run No.	Mean Particle Dia., cm	Drying Temp., Deg. C	Potassium Content (per g MAF coal basis), mg		Potassium Removed (normalised), %
			CAR	HWD Coal	
4	0.4699	282.5	0.770	1.50	4.52
5	0.4699	296	0.990	0.660	13.50
6	0.4699	335	0.990	0.780	10.40
7	0.4699	305	0.880	0.900	8.08
8	0.4699	311	0.880	0.680	6.75
9	0.4699	262.5	1.00	0.110	37.80
10	0.4699	327	1.30	1.30	4.04
11	0.4699	360	1.30	1.20	6.14
12	0.0505	325	2.67	3.18	2.36
13	0.0505	340	2.67	2.57	2.64
14	0.0505	313	2.67	2.69	3.36
15	0.0505	348	3.20	2.62	3.46
16	0.0505	298	3.20	2.90	2.81
17	0.0505	313	3.20	3.20	2.56
18	0.0505	328	2.45	3.44	2.43
19	0.0505	303	2.45	2.96	0.404
20	0.0505	343	2.45	2.73	0.843

TABLE 18

## Magnesium Removal on Hot Water Drying

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Magnesium Content (per g MAF coal basis), mg		Magnesium Removed (normalised), %
			CAR	HWD Coal	
4	0.4699	282.5	1.80	1.90	5.01
5	0.4699	296	2.70	2.50	2.40
6	0.4699	335	2.70	2.20	2.31
7	0.4699	305	3.28	2.96	2.23
8	0.4699	311	3.28	2.70	1.33
9	0.4699	262.5	2.40	2.70	4.40
10	0.4699	327	2.64	2.90	3.29
11	0.4699	360	2.64	3.13	0.808
12	0.0505	325	3.66	5.14	0.455
13	0.0505	340	3.66	4.33	0.519
14	0.0505	313	3.66	7.98	0.305
15	0.0505	348	3.40	3.80	1.40
16	0.0505	298	3.40	2.80	1.63
17	0.0505	313	3.40	3.60	0.875
18	0.0505	328	3.60	4.40	0.863
19	0.0505	303	3.60	3.50	1.13
20	0.0505	343	3.60	3.50	0.785

TABLE 19

## Calcium Removal on Hot Water Drying

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Calcium Content (per g MAF coal basis) mg		Calcium Removed (normalised), %
			CAR	HWD Coal	
4	0.4699	282.5	15.9	14.0	6.33
5	0.4699	296	19.7	18.4	4.00
6	0.4699	335	19.7	15.0	5.73
7	0.4699	305	26.9	24.1	3.82
8	0.4699	311	26.9	23.4	3.71
9	0.4699	262.5	17.1	17.5	5.10
10	0.4699	327	19.9	23.6	5.23
11	0.4699	360	19.9	23.7	9.08
12	0.0505	325	23.2	26.8	6.65
13	0.0505	340	23.2	23.7	0.00
14	0.0505	313	23.2	47.2	3.37
15	0.0505	348	28.7	19.9	7.36
16	0.0505	298	28.7	17.2	8.04
17	0.0505	313	28.7	21.2	35.80
18	0.0505	328	24.2	18.6	7.80
19	0.0505	303	24.2	14.6	9.81
20	0.0505	343	24.2	15.6	10.50



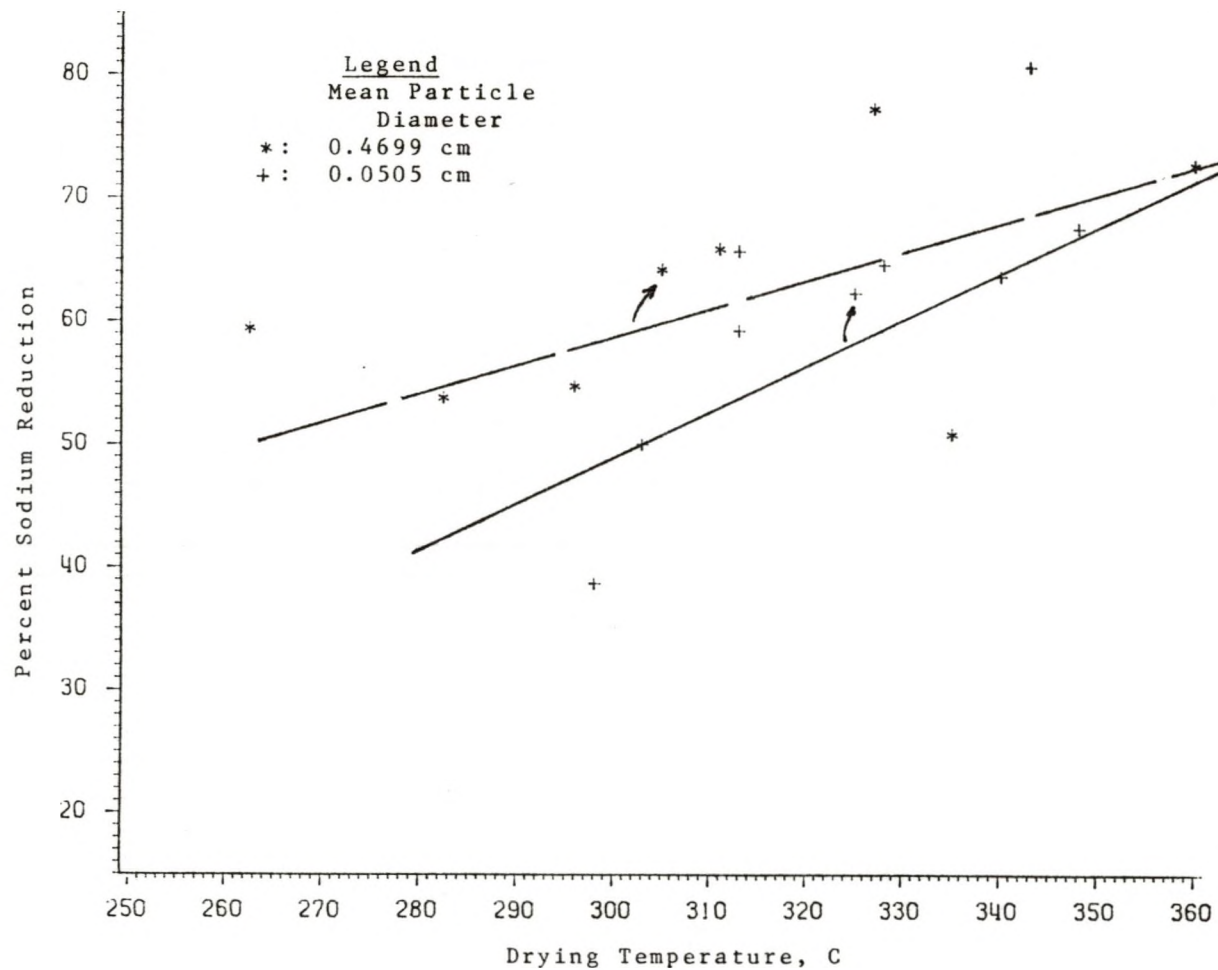


Figure 12: Sodium Reduction (normalized) as a Function of Drying Temperature

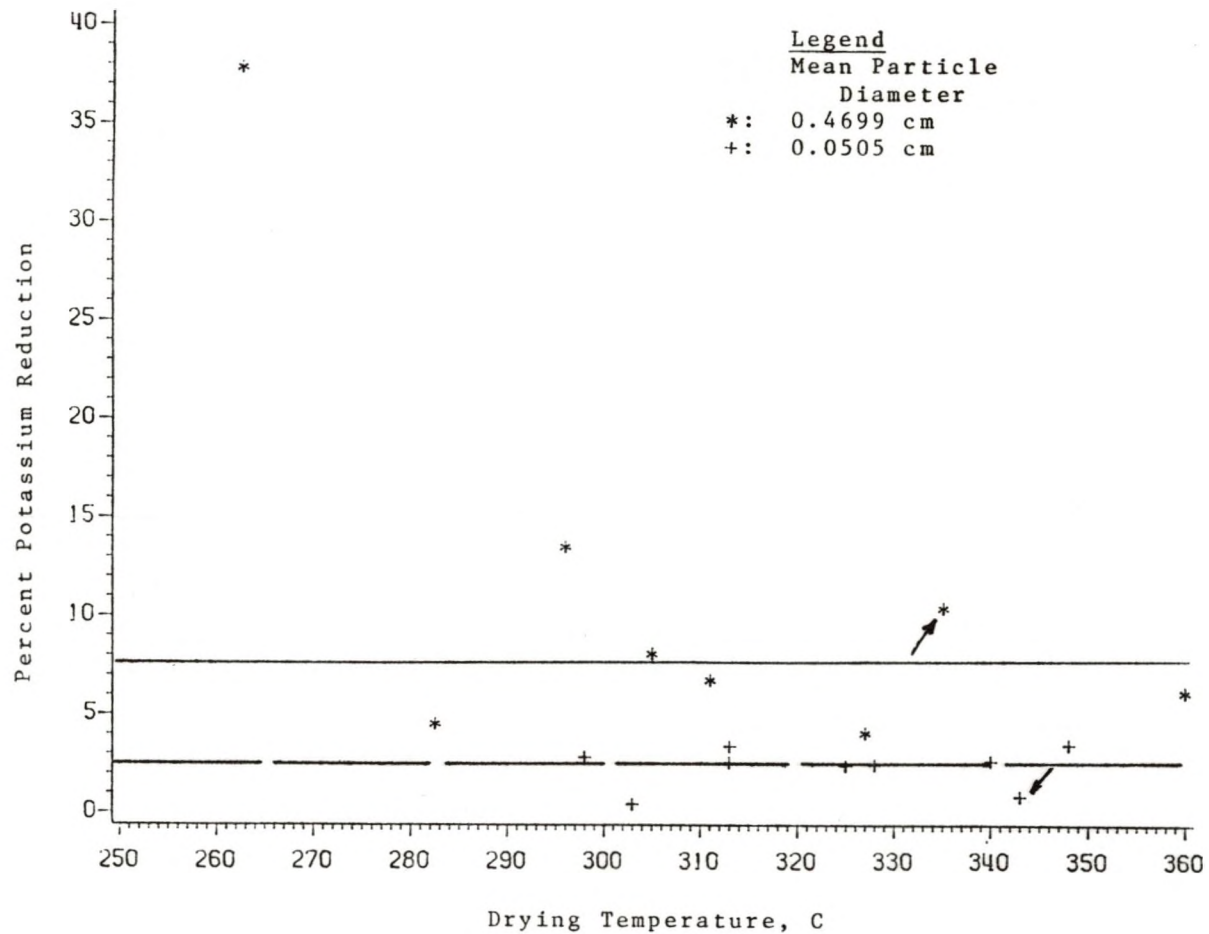


Figure 13: Potassium Reduction (normalized) as a Function of Drying Temperature

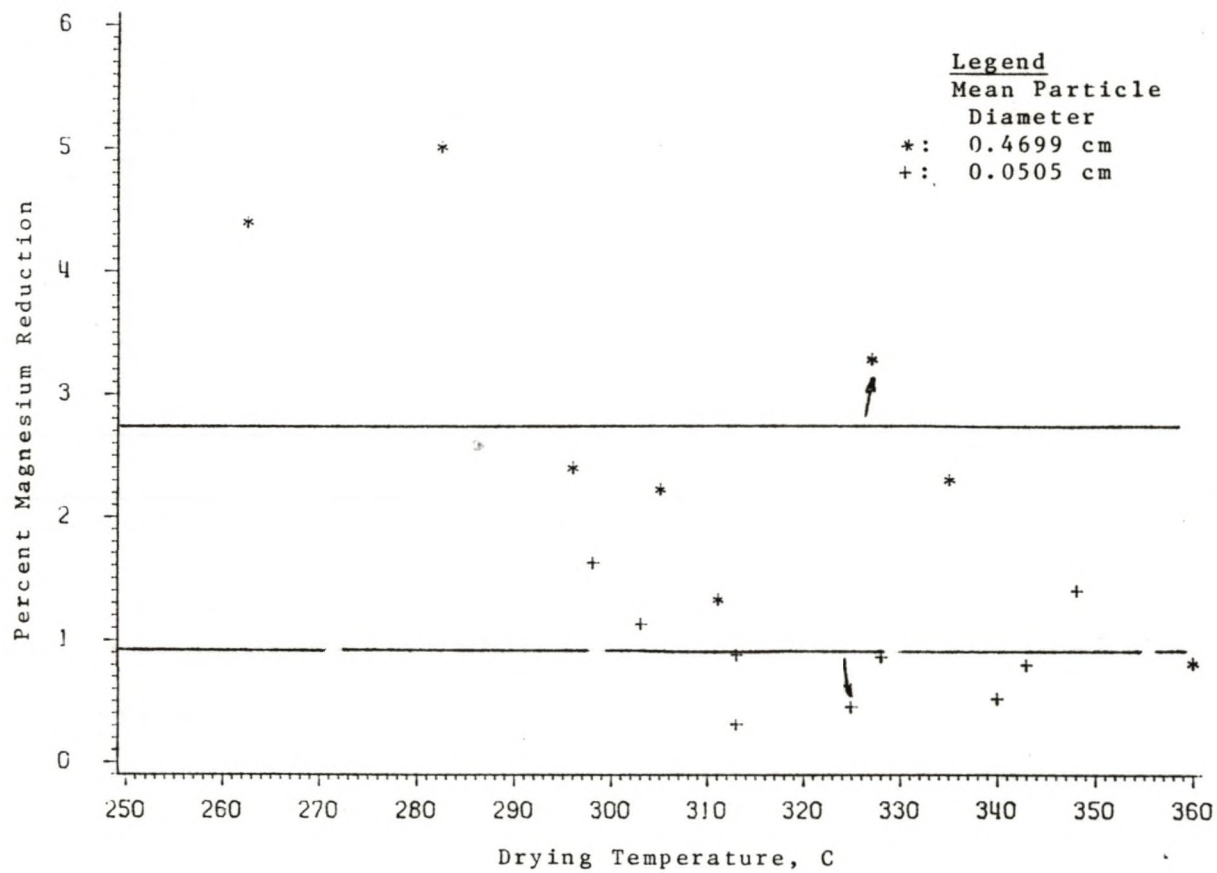


Figure 14: Magnesium Reduction (normalised) as a Function of Drying Temperature



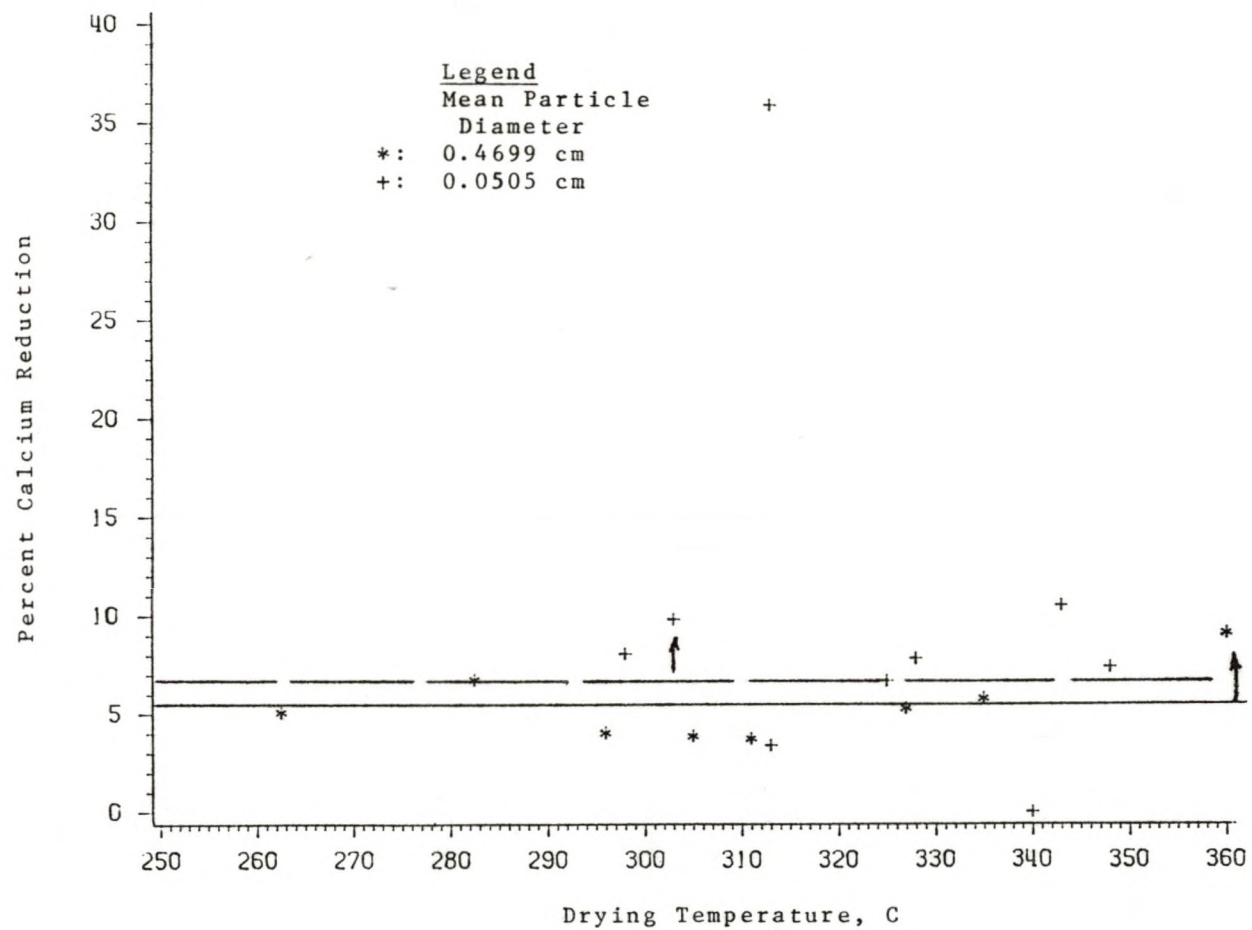


Figure 15 : Calcium Reduction (normalised) as a Function of Drying Temperature

EFFECT OF DRYING TEMPERATURE ON ACID GROUPS REMOVAL

Table 20 shows the acid groups content and percent acid groups reduction for 0.4699 cm and 0.0505 cm mean particle diameter coals as a function of drying temperature. The carboxylic acid groups reduction on hot water drying coal was plotted as a function of drying temperature in Figure 16. Figure 17 shows the carboxylic acid groups content of hot water dried coal as a function of drying temperature.

TABLE 20

Reduction of Acid Groups Content on Hot Water Drying

Run No.	Mean Particle Dia., cm	Drying Temp., deg. C	Milliequivalents of Acid Present Meq/g dry coal		Reduction of Acid Groups, %
			CAR	HWD Coal	
4	0.4699	282.5	3.91	3.31	26.3
5	0.4699	296	4.15	2.83	42.2
6	0.4699	335	4.15	2.56	45.6
7	0.4699	305	3.91	2.22	52.9
8	0.4699	311	3.91	1.31	71.5
9	0.4699	262.5	5.30	4.77	15.0
10	0.4699	327	4.76	2.28	58.0
11	0.4699	360	4.76	1.50	73.6
12	0.0505	325	3.68	1.08	73.9
13	0.0505	340	3.68	1.02	75.6
15	0.0505	348	3.50	0.693	83.8
16	0.0505	298	3.50	2.39	40.4
17	0.0505	313	3.50	1.86	52.7
18	0.0505	328	3.45	1.40	67.3
19	0.0505	303	3.45	2.54	35.6
20	0.0505	343	3.45	1.23	69.4

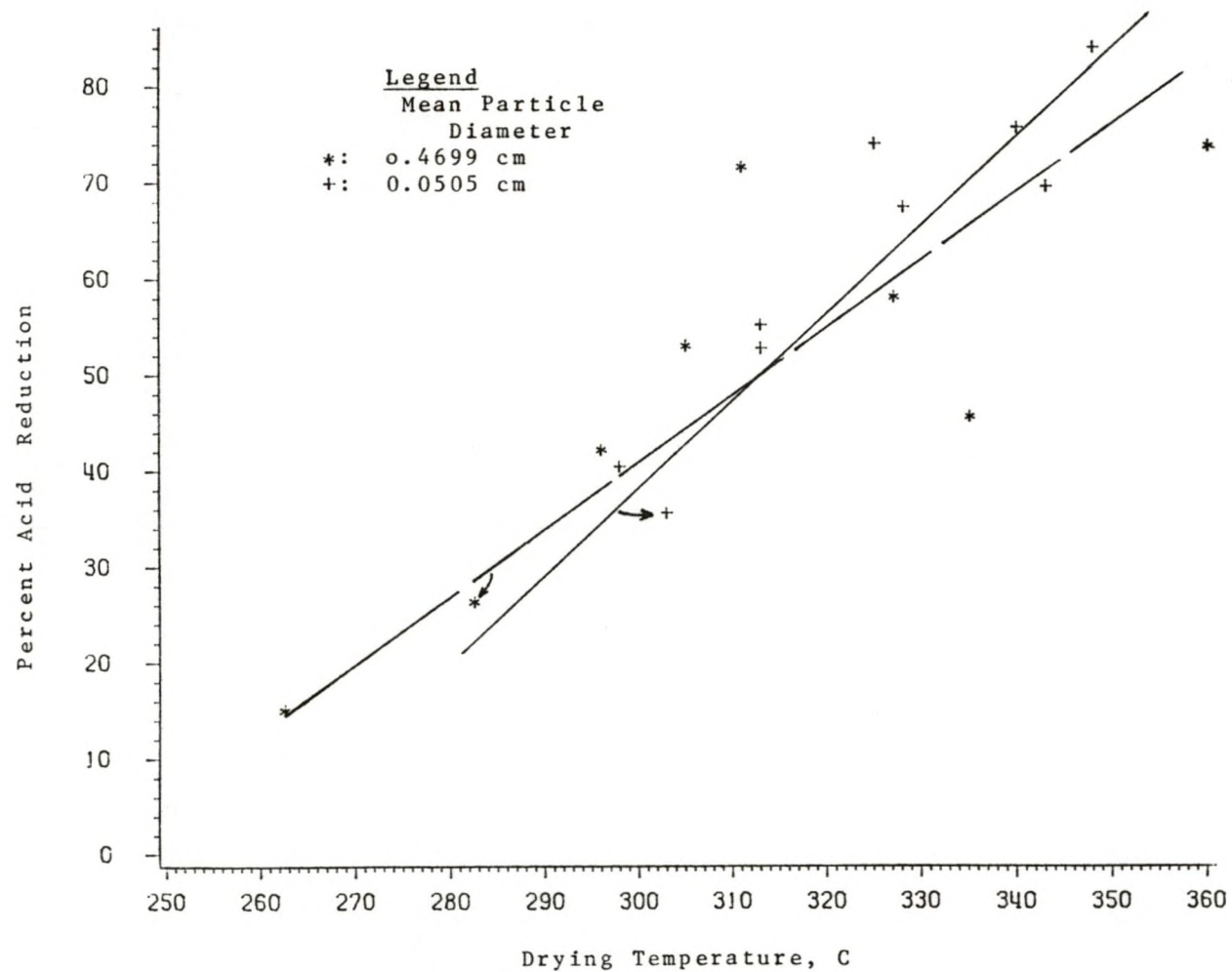


Figure 16 : Reduction in Acid Groups Content as a Function of Drying Temperature



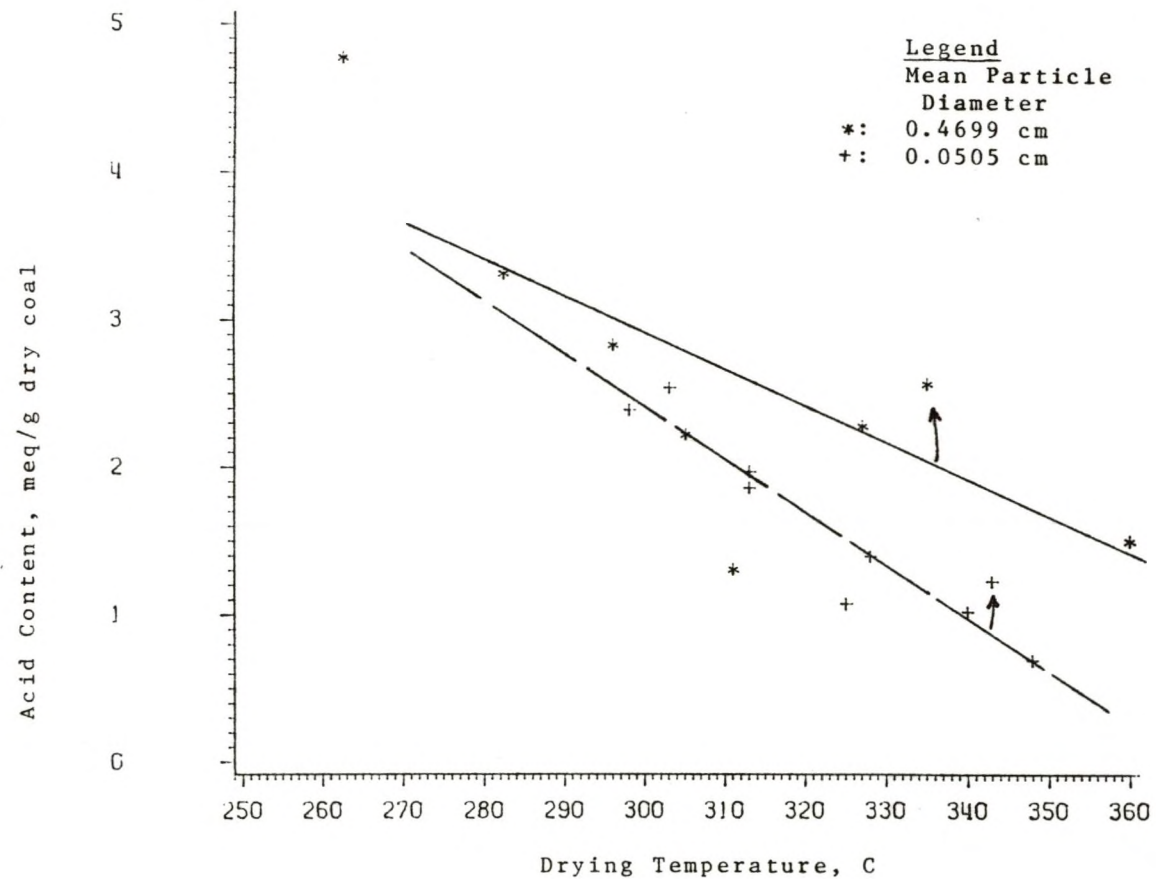


Figure 17: Acid Groups Content of Hot Water Dried Coal  
as a Function of Drying Temperature

The acid groups content of the coal decreased with increase in drying temperature upon hot water drying. For both the particle sizes, the reduction in acid groups increases linearly with the drying temperature. Also, the reduction in acid groups was sensitive to drying temperature in that as the drying temperature is increased, reduction in acid groups increased rapidly. This is also apparent from the large value of the slopes of the graphs of acid groups reductions versus drying temperature in Figure 16 for both particle sizes. There is apparently no effect of particle size on acid groups reduction. Appendix J show sample calculations for acid groups content and acid groups removal on hot water drying.

Table 21 shows the carbon dioxide content of the residual gas as a function of drying temperature for both particle sizes. Figure 18 is a plot of the carbon dioxide content of the residual gas as a function of drying temperature. It is seen that carbon dioxide content also increases as the drying temperature is increased. This was seen to be true for both the particle sizes, and there was no significant effect of particle size on carbon dioxide formation. Appendix K show a sample calculation for the carbon dioxide content of the residual gas.

The increase in carbon dioxide content of the residual gas with increase in drying temperature is consistent with

TABLE 21

## Carbon Dioxide Content in Residual Gas

Run No.	Mean Particle Dia. cm	Drying Temp., deg. C	Mole % Carbon Dioxide in Residual Gas		Moles of Carbon Dioxide in Residual Gas, (per 100 g dry coal)
			Normalised	Normalised (air free)	
4	0.4699	282.5	83.00	95.00	0.041
5	0.4699	296	85.40	95.60	0.045
6	0.4699	335	85.00	94.80	0.378
7	0.4699	305	82.60	95.20	0.047
8	0.4699	311	85.80	95.60	0.060
9	0.4699	262.5	78.80	94.50	0.038
10	0.4699	327	82.80	95.00	0.043
11	0.4699	360	80.80	92.50	0.075
12	0.0505	325	85.80	96.80	0.072
13	0.0505	340	90.50	95.50	0.101
14	0.0505	313	87.90	96.70	0.067
15	0.0505	348	88.90	96.20	0.082
16	0.0505	298	89.10	97.20	0.057
17	0.0505	313	79.90	95.60	0.055
18	0.0505	328	61.70	98.50	0.045
19	0.0505	303	84.00	95.80	0.057
20	0.0505	343	07.13	100	0.006

the decrease in carboxylic acid groups with increase in drying temperature. The carbon dioxide is formed by the break up of the carboxylic groups.

The main product of thermal decomposition of acid groups (carboxylic functional groups) in brown coal and lignite is carbon dioxide. Approximately 4/5ths of the carbon dioxide recorded comes from the acid groups (26). Hence, as the acid group decomposition increases with temperature, the



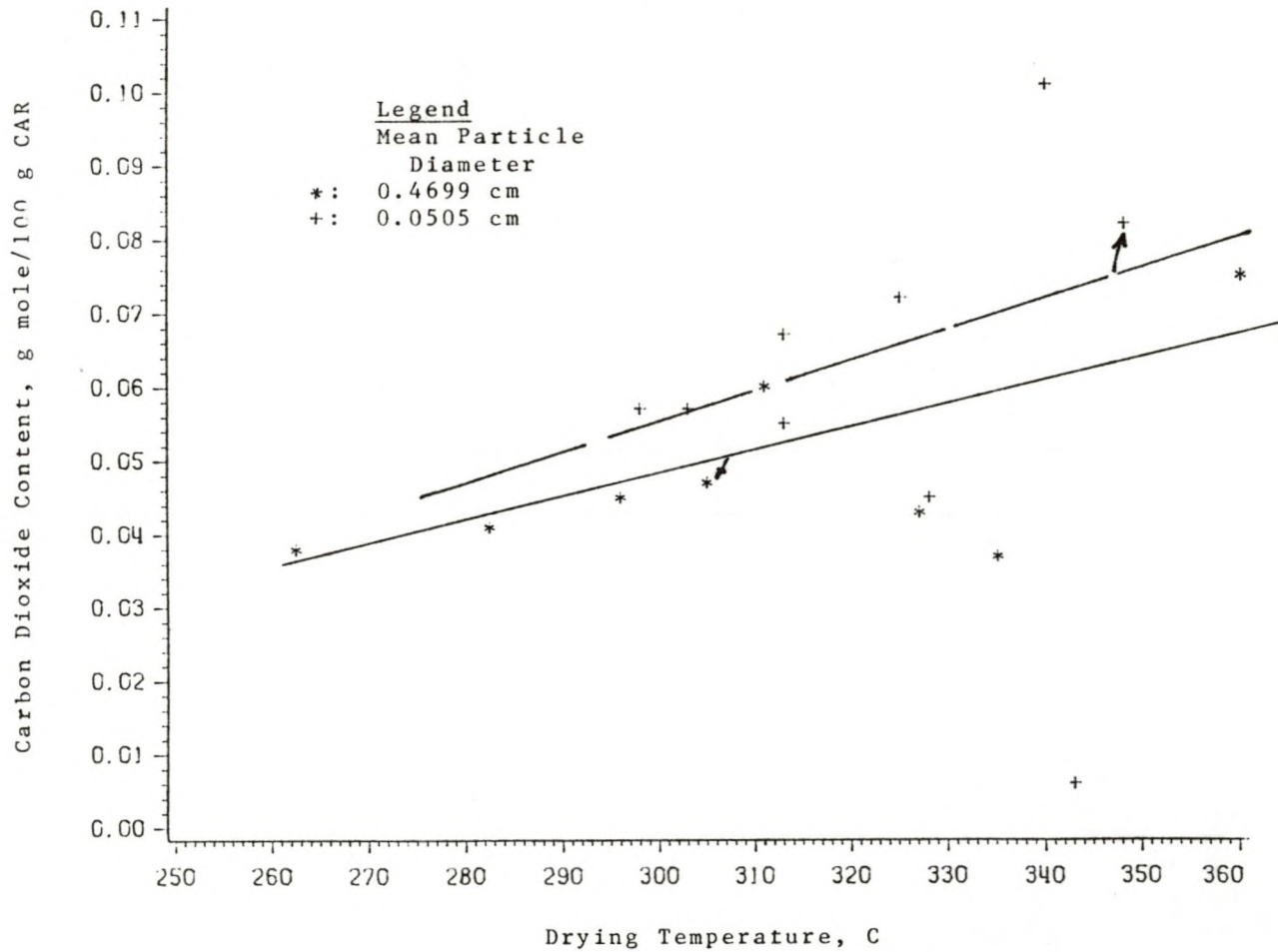


Figure 18: Carbon Dioxide Content in Residual Gas as a Function of Drying Temperature

amount of carbon dioxide formed in the residual gas should also increase. Similar results were obtained by Maas (29).

In the present study, no attempt was made to calculate the equivalent amounts of phenolic groups and carboxyl groups from the total amount carbon dioxide and carbon monoxide formed in the residual gas. This was because no reasonable assumption could be made for the calculations. Further investigations regarding the sources of the various thermal decomposition products of the coal studied should be carried out before attempts, to calculate equivalent amounts of carboxylic and phenolic groups from their thermal decomposition products, are made.

If the present study is modified to treat a coal-water slurry in a nitrogen atmosphere, and specific functional groups are tested, then evidence showing how the functional groups decompose in the coal used in this study would become apparent. The metal ions, sodium, potassium, magnesium, and calcium interfere in the analysis for carboxylic groups determination since they are present as carboxylates. Hence, their removal prior to analysis for acid groups, could give a better determination of the acid groups content. Schafer (14) in his work had removed the cations like, sodium, magnesium, and calcium from the coal by acid extraction prior to the acid groups determinations.

EFFECT OF DRYING TEMPERATURE ON CALORIFIC VALUE OF COAL

The calorific or heating values of the original and hot water dried coals are tabulated in Table 22 for the various drying temperatures for the two particle sizes. The heating value of the dried coal is plotted in Figure 19 as a function of the drying temperature. The calorific value of the hot water dried coal increased upon hot water drying with the increase in drying temperature for both the particle sizes. The average calorific value for the as recieved coal was 5973 cal/g dry coal (for 0.4699 cm mean diameter particles) and 5461 cal/g dry coal (for 0.0505 cm mean diameter particles).

It was noticed that there was a significant effect of particle size on calorific value. Larger particles had a significantly higher calorific value than the smaller particles. As the carboxylic groups in the coal break down upon hot water drying, carbon dioxide is released, decreasing the oxygen content of the coal, and consequently producing a coal of higher heating value. It has been shown that as the drying temperature is increased, there is increased breakdown of carboxylic groups, and hence it is consistent with the increase in the heating value of the hot water dried coal. Maas (29) reported a linear increase in heating value of hot water dried lignite. Appendix L show a sample calculation for the calorific value of coal.



TABLE 22

Calorific Value of CAR and HWD Coal

Run No.	Mean Particle Dia., cm	Drying Temperature, deg. C	Calorific Value, cal/g dry coal	
			CAR	HWD Coal
4	0.4699	282.5	5,901	6,122
5	0.4699	296	6,008	6,427
6	0.4699	335	6,008	6,480
7	0.4699	305	5,855	6,484
8	0.4699	311	5,855	6,354
9	0.4699	262.5	6,466	6,832
10	0.4699	327	5,843	6,427
11	0.4699	360	5,843	6,431
12	0.0505	325	5,624	6,054
13	0.0505	340	5,624	6,106
14	0.0505	313	5,624	5,817
15	0.0505	348	5,360	6,066
16	0.0505	298	5,360	5,714
17	0.0505	313	5,360	5,601
18	0.5050	328	5,388	5,618
19	0.0505	303	5,388	5,542
20	0.0505	343	5,388	5,736

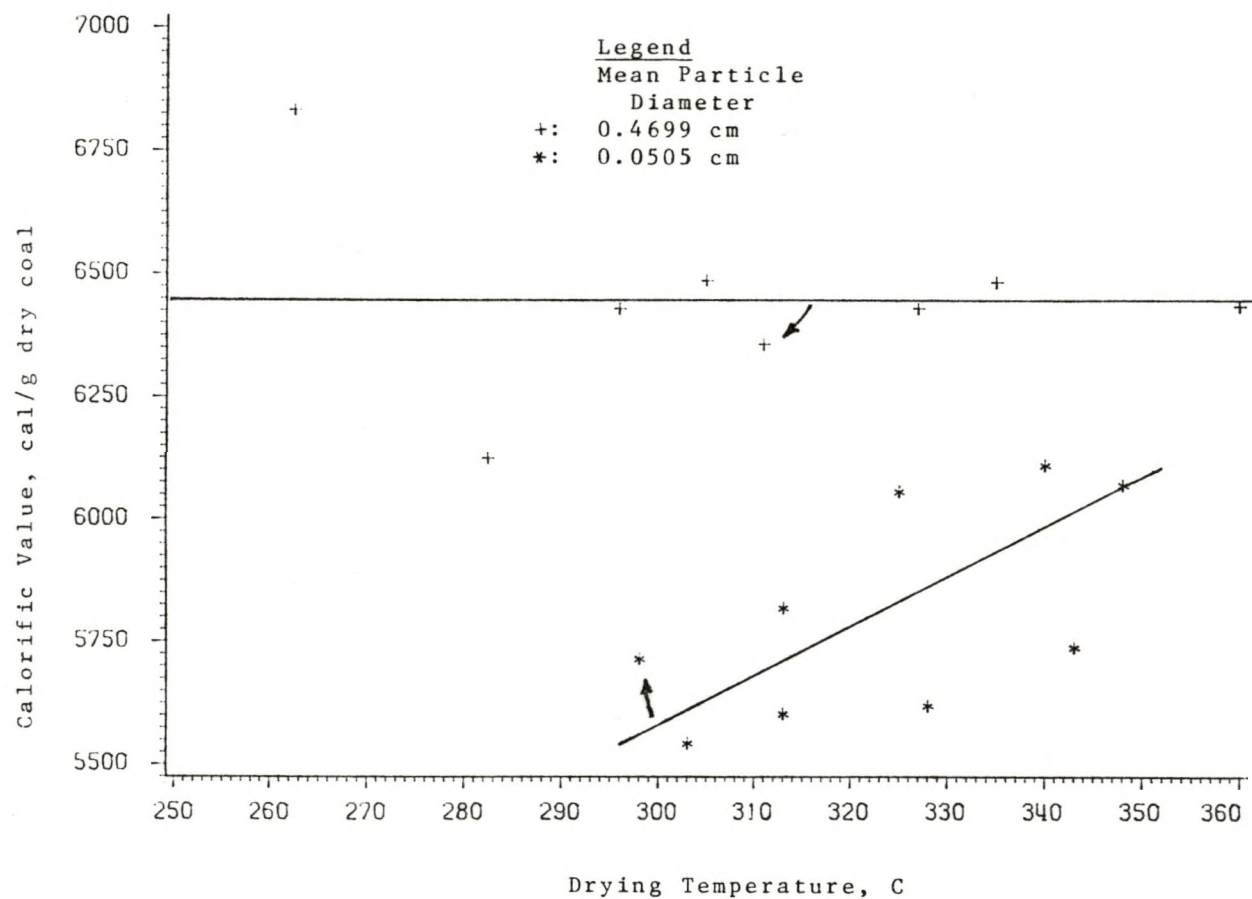


Figure 19: Calorific Value of Hot Water Dried Coal as a Function of Drying Temperature

OVERALL MATERIAL BALANCE CLOSURE

Overall material balance closures were calculated for each of the 17 runs. They are tabulated in Table 23 for various temperatures for the two particle sizes. Total material balance closures vary between 91% and 106% for all the runs. Mass of residual gas formed was neglected for the purpose of total material balance, because it was negligible (of the order of 2 to 3 g) compared to the total mass of slurry processed. Mass of gas formed was around 1 % of the total mass of slurry processed. Maas (29) used the same autoclave with which this study was done, for his investigation of the hot water drying characteristics of a North Dakota lignite. He found from blank runs that 10 g of water was permanently lost for each run in the piping section of the autoclave and was unrecoverable. This fact accounts for the lower overall material balances for the autoclave runs made in this study.



TABLE 23

## Overall Material Balance

Run No.	Mean Particle Dia., cm	Drying Temperature, deg. C	Overall Material Balance Closure, %
4	0.4699	282.5	96.3
5	0.4699	296	93.6
6	0.4699	335	93.1
7	0.4699	305	94.4
8	0.4699	311	94.6
9	0.4699	262.5	105.7
10	0.4699	327	91.0
11	0.4699	360	94.4
12	0.0505	325	95.7
13	0.0505	340	96.1
14	0.0505	313	97.4
15	0.0505	348	96.0
16	0.0505	298	94.6
17	0.0505	313	95.5
18	0.0505	328	91.8
19	0.0505	303	96.6
20	0.0505	343	96.0

EFFECT OF DRYING TEMPERATURE ON RESIDUAL GAS COMPOSITION

Table 24 shows the normalised, air free composition of the residual gas formed in the autoclave during the run. Since the autoclave contains some air at the start of the run, an air free composition gives a clearer picture of the relative amounts of the constituent gases present in the residual gas. The table is arranged in increasing order of drying temperature for both particle sizes.

TABLE 24

Residual Gas Composition (normalised, air free)

Mean Particle Dia., cm	Drying Temp. deg. C	Normalised, Air Free, Composition, Mole %						
		CO <sub>2</sub>	C <sub>2</sub> H <sub>6</sub>	H <sub>2</sub>	CH <sub>4</sub>	CO	H <sub>2</sub> S	NH <sub>3</sub>
0.4699	262.5	94.50	0.02	0.24	0.83	4.38	0.04	0.0
0.4699	282.5	95.02	0.03	0.09	1.04	3.72	0.08	0.01
0.4699	296	95.64	0.04	0.43	1.03	2.81	0.04	0.0
0.4699	305	95.20	0.08	0.21	1.64	2.83	0.03	0.01
0.4699	311	95.61	0.10	0.16	1.66	2.44	0.0	0.0
0.4699	327	95.00	0.11	0.16	1.49	3.23	0.0	0.0
0.4699	335	94.78	0.07	0.13	1.12	3.29	0.57	0.04
0.4699	360	92.54	0.55	0.84	4.16	1.80	0.0	0.0
0.0505	298	97.16	0.0	0.09	0.38	2.37	0.0	0.0
0.0505	303	95.84	0.0	0.42	0.71	2.99	0.0	5.04
0.0505	313	95.63	0.0	0.57	1.03	2.77	0.0	0.0
0.0505	313	96.68	0.04	0.18	0.79	2.31	0.0	0.0
0.0505	325	96.77	0.05	0.21	1.24	1.54	0.19	0.0
0.0505	328	98.51	0.0	0.50	0.98	0.0	0.0	0.0
0.0505	340	95.50	0.16	0.41	1.39	1.98	0.44	0.11
0.0505	343	99.99	0.0	0.0	0.0	0.0	0.0	0.0
0.0505	348	96.21	0.22	0.25	1.27	2.06	0.0	0.0

The carbonyl groups are stable upto 150 C, but decompose at higher temperatures to form carbon monoxide. Hence, larger amounts of carbon monoxide may be expected with increasing temperatures. The amount of carbon monoxide formed in the residual gas, in terms of mole percent, appears to decrease with the increase in drying temperature for both particle sizes. This is probably because of the relative amounts of other gases formed as well. The total moles of carbon monoxide formed would probably be a better basis for determining the effect of drying temperature on the amount

of carbon monoxide formed. As can be seen from the table that the quantity of hydrogen sulfide formed in the residual gas is almost negligible compared to the quantity of carbon dioxide and carbon monoxide formed, and in most cases runs undetectable. Sulfur is present in the residual gas as hydrogen sulfide. Methane content of the residual gas is also low. As the drying temperature increases, the carbon dioxide content, the ethane content, and methane content of the residual gas also increase. The drying temperature does not significantly effect the hydrogen content of the residual gas. The carbon monoxide content of the residual gas decrease as the drying temperature increases. These results are apparent from the Table 24.



## Chapter VII

### CONCLUSION AND RECOMMENDATION

#### CONCLUSION

1. Moisture reduction was found to be independent of the drying temperature for both the particle sizes.
2. Particle size did not have any significant effect on moisture reduction.
3. Ash content did not change significantly due to hot water drying for both particle sizes.
4. Although the equilibrium moisture content decreased with increase in drying temperature, the smaller particles exhibited a higher equilibrium moisture content.
5. Sodium reduction increased with drying temperature for both particle sizes, but there was no significant effect of particle size on sodium reduction.
6. Sulfur removal on hot water drying was low for both the particle sizes.
7. Acid groups decomposition, calorific value of the hot water dried coal, and carbon dioxide formation increased as the drying temperature increased.
8. As the drying temperature increased, the ethane content and the methane content of the residual gas increased.

9. Drying temeprature had no influence on the hydrogen content of the residual gas.
10. The carbon monoxide content of the residual gas decreased as the drying temperature increased.

#### RECOMMENDATION

1. Runs should be performed with particles of mean particle diameter other than 0.0505 cm and 0.4699 cm to determine if the conclusions arrived at in the present study are valid over other particle sizes.
2. Slurry concentration and residence times could be varied to determine their effect on moisture reduction, ash content, sulfur and sodium removal, acid groups reduction and calorific value.

## Appendix A

### DETERMINATION OF MOISTURE AND ASH OF COAL

The moisture and ash contents of the CAR and the hot water dried coals were calculated according to the usual ASTM procedures #D3173 and #D3174 respectively (32). The following is a sample calculation of the moisture content and ash content of a sample of coal.

#### SAMPLE DATA FOR RUN #8

1. Type of coal :	Hot water dried coal	
2. Sample:	A	B
3. Mass of cruc. =	13.1799 g	12.1320 g
4. Mass of cruc. + sample =	15.1031 g	14.0675 g
5. Mass of sample =	1.9232 g	1.9355 g
6. Mass of cruc.+ dried sample=	14.8521 g	13.8091 g
7. Mass of dried sample =	1.6722 g	1.6771 g
8. Mass of cruc. + ash =	13.3913 g	12.3413 g
9. Mass of ash =	0.2114 g	0.2093 g
10. Percent moisture =	13.00 %	13.30 %
11. Average percent moisture =	13.20 %	
12. Percent ash (wet) =	11.00 %	10.80 %
13. Average percent ash (wet) =	10.90 %	
14. Average percent ash (dry) =	12.60 %	



The values in the first column are that of the first sample and the values in the second column are that of the duplicate sample of the hot water dried coal.

#### SAMPLE CALCULATION FOR MOISTURE AND ASH CONTENTS

Percent Moisture is calculated as follows:

Percent Moisture

$$= \frac{(\text{Mass of sample} - \text{Mass of oven dried sample}) \times 100}{\text{Mass of sample}}$$

Substituting values into the above formula we get

$$\text{Percent Moisture} = \frac{(1.9232 - 1.6722) \text{ g}}{(1.9232) \text{ g}} \times 100 = 13.00 \%$$

Percent ash content (wet basis) is calculated as follows :

$$\text{Percent ash (wet basis)} = \frac{(\text{Mass of ash})}{(\text{Mass of sample})} \times 100$$

Substituting values in the above formula we get

$$\text{Percent ash} = \frac{0.2114 \text{ g}}{1.9232 \text{ g}} \times 100 = 11.00 \%$$

Percent ash content (dry basis) is calculated as follows :

$$\text{Percent ash (dry basis)} = \frac{(\text{Mass of ash}) \times 100}{(\text{Mass of dried sample})}$$

or,

$$\text{Percent ash (dry basis)} = \frac{\text{Mass of ash} \times 100}{(\text{Mass of sample})(1 - \text{Fractional moisture content})}$$

Average percent ash (dry basis)

$$= \frac{10.90 \%}{(1 - 0.1320)} = 12.60 \%$$

where 10.90 % and 0.1320 are the percent ash (wet) and fractional moisture content in the coal sample respectively. The moisture and ash contents of CAR was calculated in a similar way.

## Appendix B

### CALCULATION OF THE AMOUNT OF MOISTURE INTAKE (ALSO SURFACE MOISTURE) BY ORIGINAL COAL ON WETTING WITH WATER

Coals of different particle sizes absorb different amounts of water on wetting. Experiments were performed to determine the amount of water intake on wetting.

#### SAMPLE DATA FROM COAL WETTING EXPERIMENTS FOR 0.4699 CM MEAN DIAMETER ORIGINAL COAL PARTICLES

- (i) Moisture content of the CAR = 21.7 %
- (ii) Moisture content of wetted CAR (after 1/2 hr of wetting; no excess moisture) = 25.61%
- (iii) Moisture content of wetted CAR (after 2 hr of wetting; no excess moisture) = 25.65 %
- (iv) Moisture content of wetted CAR (after 24 hr of wetting; no excess moisture) = 24.90 %

As can be seen from the above values, the moisture content of the wetted coals does not vary much and hence was considered to be a constant. Therefore, an average value of moisture content was taken.

The average moisture content of the 3 samples (1/2 hr, 2 hr, and 24 hr) of the wetted coal (no surface moisture) = 25.49 %.



SAMPLE CALCULATION OF WATER INTAKE BY 0.4699 CM MEAN  
DIAMETER ORIGINAL COAL PARTICLES ON WETTING WITH WATER

(i) Initial moisture of CAR = 21.70 %

(ii) Final moisture after wetting CAR

(no excess moisture) = 25.49 %

Water intake on wetting =  $A/(100-A) - B/(100-B)$

where,

A = Final percent moisture content of wetted CAR

B = Initial percent moisture content of CAR

On substitution of the values we get moisture intake on wetting

$$\begin{aligned} &= 25.49/(100-25.49) - 21.70/(100-21.70) \text{ g water/g MF coal} \\ &= 0.0649 \text{ g water/g MF coal} \end{aligned}$$

This is the factor to be subtracted from the moisture content of the 0.4699 cm mean diameter hot water dried coal particles wiped off surface moisture, to obtain the inherent moisture content of the hot water dried coal. Water intake on wetting is also the surface moisture of the coal due to wetting. Similarly the moisture intake by 0.0505 cm mean diameter original coal particles was found out.

To have an idea of the trend of moisture intake with respect to the particle size, the experiment was repeated with two other particle sizes. It was noticed that as the mean particle diameter of the original coal increased the moisture intake by the original coal particles decreased. Table 25 and Figure 20 show the results of these coal wetting experiments.

TABLE 25

Moisture Intake on Wetting CAR

Mean Particle Dia., cm	Moisture Intake, g moisture/g MF coal
0.0505	0.2521
0.1001	0.2240
0.2189	0.1800
0.4699	0.0649

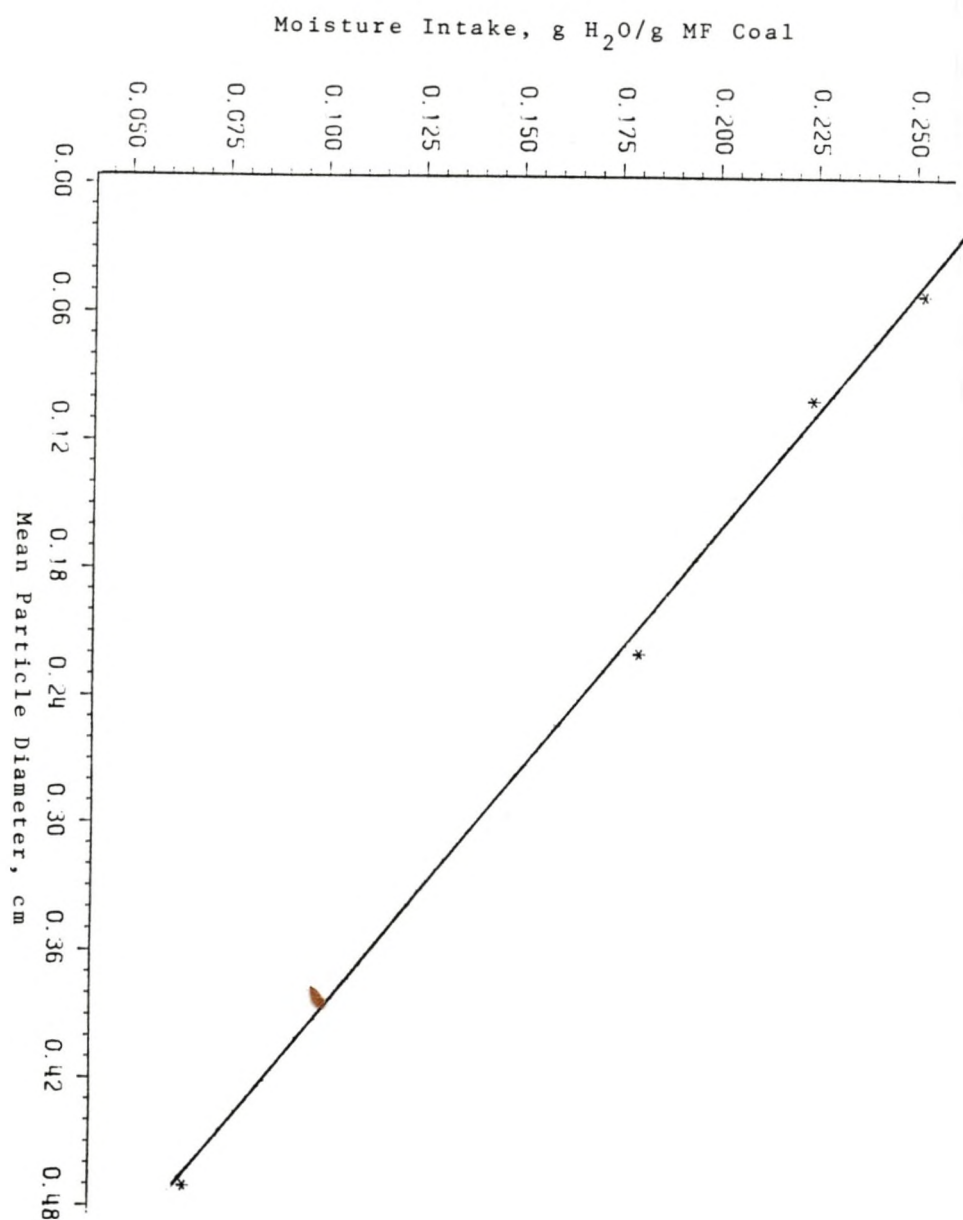


Figure 20: Moisture Intake by CAR Particles on Wetting With Water



## Appendix C

### CALCULATION OF THE INHERENT MOISTURE CONTENT OF HOT WATER DRIED COAL AND THE PERCENT MOISTURE REMOVAL ON HOT WATER DRYING

Hot water dried coal contains inherent as well as surface moisture. The inherent moisture in the original coal is removed during the hot water drying process, and hence the inherent moisture is used in the calculation of the moisture reduction. Sample calculation of the inherent moisture of a sample of hot water dried coal and percent moisture reduction on hot water drying of a sample run is as follows.

#### SAMPLE CALCULATION OF THE INHERENT MOISTURE CONTENT OF THE OF THE HOT WATER DRIED COAL

##### Sample Data for Run #8

- (i) Mean Particle Diameter = 0.4699 cm
- (ii) Percent moisture of HWD coal = 13.20 %
- (iii) Percent moisture of CAR = 23.60 %
- (iv) Moisture intake on wetting = 0.0649 g moisture/g MF coal (from Table 25).

##### Calculation of the Inherent Moisture Content

The inherent moisture content of the HWD coal (based on MF coal)

$$= (\text{moisture content of the HWD coal} - \text{moisture}$$

intake on wetting) based on MF coal On  
 substituting the values, inherent moisture of the HWD coal  

$$= (0.1521 - 0.0649) \text{ g moisture/g MF coal}$$

$$= 0.0872 \text{ g moisture/g MF coal}$$

Hence, the inherent moisture content of the HWD coal, on a  
 wet basis =  $(0.0872)(100)/(1+0.0872) = 8.02 \%$

#### SAMPLE DATA FOR RUN #8

- (i) Percent moisture of CAR = 23.60 %
- (ii) Amount of original coal processed = 147 g
- (iii) Percent moisture of HWD coal (no excess moisture) =  
13.20 %
- (iv) Amount of HWD coal obtained = 110.7 g
- (v) Percent inherent moisture of HWD coal = 8.02 %

#### SAMPLE CALCULATION FOR MOISTURE REMOVAL

Amount of moisture in the CAR processed,  $(A) = (147)(0.236)$   

$$= 34.7 \text{ g}$$

Amount of moisture free HWD coal =  $(110.7)(1 - 0.132)$   

$$= 96.1 \text{ g}$$

Amount of inherent moisture in HWD coal,  $(B) = (96.1)(0.0872)$   

$$= 8.38 \text{ g}$$

The percent moisture removal =  $(A - B)(100)/(A)$

where A and B are defined above

Substituting the values, the percent moisture reduction

$$= \frac{(34.7 - 8.38)(100)}{(34.7)} = 75.8 \%$$

## Appendix D

### MOISTURE REABSORPTION AND DETERMINATION OF EQUILIBRIUM MOISTURE CONTENT OF ORIGINAL AND HOT WATER COALS USING A 100 % HUMIDITY CHAMBER

These moisture reabsorption tests were conducted to study the effect of drying temperature and particle size on moisture reabsorption and equilibrium moisture of the hot water dried coal.

For these tests, approximately 2 g of the coal was accurately weighed into a previously weighed watch glass. The watch glass was then placed in a 100 % humidity chamber. The watch glass was taken out of the humidity chamber every 24 hrs, off any moisture sticking to the walls of the watch glass was wiped off and weighed. This was continued till the watch glass and contents attained a constant weight. This period was approximately 16 days.

After the watch glass and contents reached a constant weight, the coal sample was removed from the watch glass and its moisture content was determined by the standard ASTM method #D3173 (32). This was the final moisture content of the sample. From the final moisture content and the final weight of the sample, the moisture content of the sample on other days were found out. Table 26 and Figures 21,22 show



the moisture content of the CAR (both 0.4699 cm and 0.0505 cm mean diameter particles) on the various days in 100 % humidity chamber. The equilibrium moistures correspond to the horizontal straight line portions of the graphs.

TABLE 26

Moisture Reabsorption Tests on CAR (both 0.4669 cm and  
0.0505 cm Mean Diameter Particles) in 100% Humidity Chamber

Time, days	Percent Moisture Reabsorption in 100% Humidity Chamber							
	CAR #4	CAR #5,6	CAR #7,8	CAR #9	CAR #10,11	CAR #12,13,14	CAR #15,16,17	CAR #18,19,20
0	18.71	21.25	22.65	19.40	20.00	22.00	21.00	21.30
1	20.76	22.67	22.10	21.92	20.16	22.94	21.75	21.94
2	20.80	22.64	21.77	22.10	20.73	23.29	22.05	22.72
3	20.87	22.64	21.45	22.22	20.90	23.78	22.52	23.28
4	20.85	22.65	21.66	22.27	21.54	23.96	22.82	23.44
5	21.00	22.85	21.71	22.39	21.83	24.00	22.95	23.48
6	21.08	22.89	21.74	22.62	22.00	24.02	23.05	23.46
7	21.14	22.90	21.77	22.71	22.10	24.02	23.05	23.50
8	21.17	22.98	21.75	22.82	22.09	24.02	23.05	23.50
9	21.03	22.85	21.77	22.71	22.10	24.03	23.05	23.50
10	21.04	22.88	21.80	22.75	22.10	24.02	23.06	23.50
11	21.06	22.90	21.83	22.79	22.10	24.05	23.05	23.48
12	21.06	22.94	21.86	22.81	22.10	24.05	23.05	23.50
13	21.19	22.95	21.66	22.84	22.08	24.05	23.05	23.50
14	21.11	22.68	21.38	23.00	22.10	24.05	23.05	23.50
16	20.96	22.67	21.31	22.70	22.10	24.04	23.05	23.50

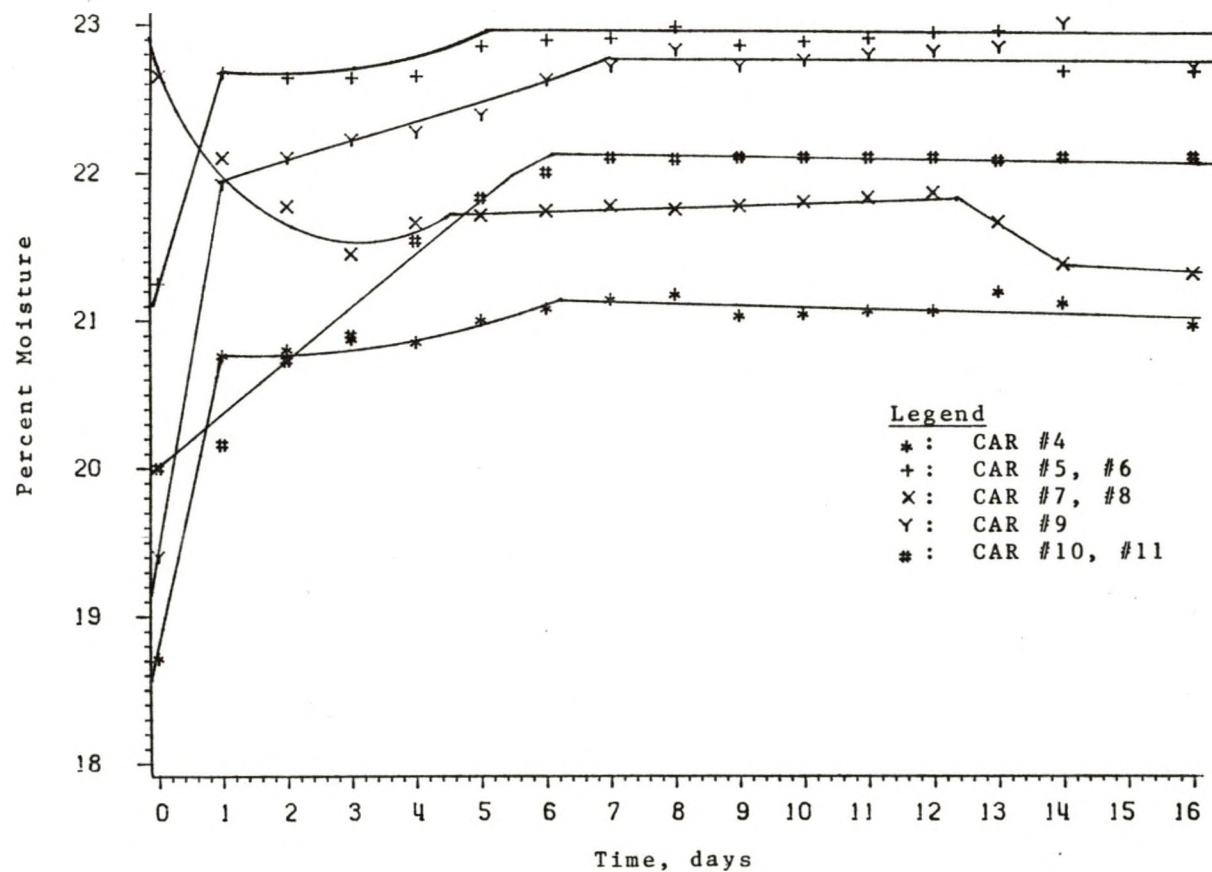


Figure 21: Moisture Reabsorption by CAR (0.4699 cm Mean Diameter Particles) in 100% Humidity Chamber



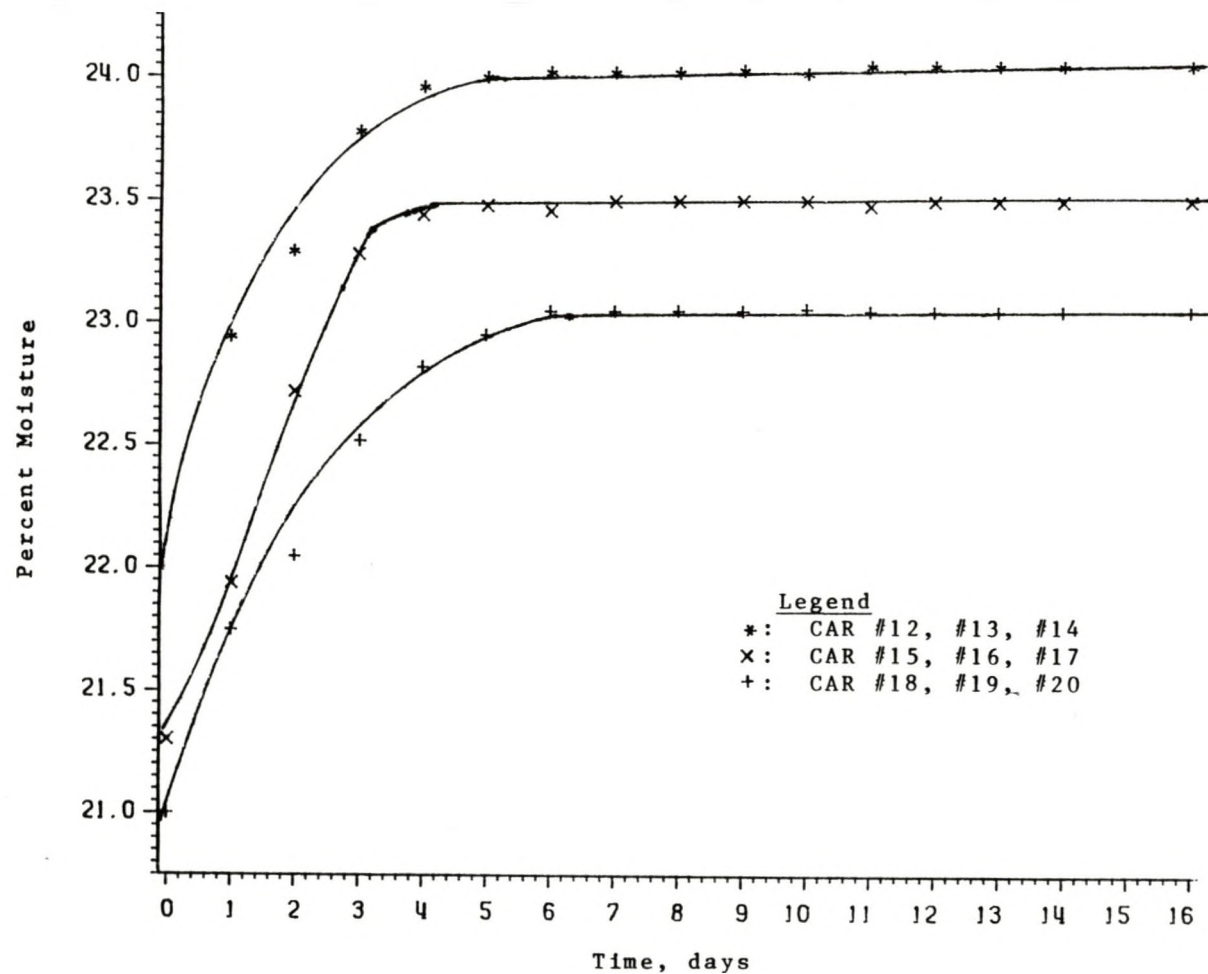


Figure 22: Moisture Reabsorption by CAR (0.0505 cm Mean Diameter Particles) in 100% Humidity Chamber

## Appendix E

### TOTAL MATERIAL BALANCE

The residual gas formed is of the order of 2-3 g. A sample calculation for the mass of residual gas formed is given below. Hence, for a total mass of coal slurry of about 300 g, the mass of gas formed is negligible. Hence, the mass of gas formed was neglected for the purpose of total material balance.

#### SAMPLE DATA FOR RUN #10

1. Mean particle diameter = 0.4699 cm
2. Slurry concentration = 1:1 (solid to liquid mass ratio)
3. Drying temperature = 327 C
4. Residence time = 16.5 min
5. Amount of CAR taken for the slurry = 144.58 g
6. Amount of water taken for the slurry = 150 g
7. Amount of slurry prepared for processing = 294.58 g
8. Amount of hot water dried slurry obtained = 268 g

SAMPLE CALCULATION FOR MASS OF RESIDUAL GAS FORMED

The following is a sample calculation for the mass of gas formed in the autoclave during run #10. For the purpose of this calculation, the residual gas formed was assumed to behave like an ideal gas and hence, assumed to obey the ideal gas law.

The ideal gas law assumed to apply was

$$PV = nRT$$

where,

P = absolute pressure in the autoclave, Kg/m<sup>2</sup>

V = volume occupied by the gas in the autoclave, m<sup>3</sup>

n = number of moles of gas in the autoclave, Kg/mole

R = universal gas constant, (m)(Kg)/(Kg/mole)(K)

T = temperature in the autoclave, K

Absolute pressure in the autoclave, P = 87646 Kg/m<sup>2</sup>

Temperature in the autoclave, T = 305 K

Universal gas constant, R = 848 (m)(Kg)/(Kg/mole)(K)

Volume occupied by the gas in the autoclave

= (total available empty space in the autoclave)

- (space occupied by the coal slurry)

= (500.5 - 280) ml

= 220.5 ml = 0.0002205 m<sup>3</sup>

Now, substituting these values in the above gas law equation we get,

$$(87646 \text{ Kg/m}^2)(0.0002205 \text{ m}^3) = (n)(848 \text{ mKg/Kgmole K})(305 \text{ K})$$

which after solving for n gives,



$$n = 0.000074721 \text{ kgmole}$$

or,

$$n = 0.074721 \text{ gmole}$$

This is the total moles of residual gas formed in the autoclave during the process. The total mass of gas formed is found as follows. Average molecular weight of the residual gas = 41.13.

Then the mass of gas formed is given by the following formula:

$$\text{Mass of gas formed} = (\text{Total moles})(\text{Avg. mol. wt.})$$

Substituting the values into the formula we get,

$$\begin{aligned} \text{Mass of gas formed} &= (0.074721 \text{ gmole})(41.13) \\ &= 3.07 \text{ g} \end{aligned}$$

#### SAMPLE TOTAL MATERIAL BALANCE CLOSURE

Total slurry in to be processed = total hot water dried slurry out

The above is true because the amount of gas formed is small and is neglected for the purpose of total material balance.

Total material balance closure

$$= (\text{Weight of slurry out})(100)/(\text{Weight of slurry in})$$

or,

Total material balance closure

$$\begin{aligned} &= (268)(100)/294.58 \\ &= 91.0 \% \end{aligned}$$

## Appendix F

### CALCULATION FOR THE SULFUR CONTENT

The following is a sample calculation for the sulfur content of a sample of CAR. The calculations for the sulfur contents of HWD coal and filtrate were done in a similar way.

#### SAMPLE DATA FOR RUN #18

- |  |           |          |
|--|-----------|----------|
| 1. Type of coal :                            | CAR       |          |
| 2. Mean particle diameter =                  | 0.0505 cm |          |
| 3. Percent moisture =                        | 28.83 %   |          |
| 4. Sample :                                  | A         | B        |
| 5. Weight of sample =                        | 0.0573 g  | 0.0711 g |
| 6. Volume of KIO3 consumed =                 | 0.035 ml  | 0.051 ml |
| 7. Therefore, percent sulfur (wet) =         | 0.679 %   | 0.797 %  |
| 8. Therefore, average percent sulfur (wet) = | 0.738 %   |          |
| 9. And, average percent sulfur (dry) =       | 1.04 %    |          |

The values in the first column are that of the first sample and the values in the second column are that of the duplicate sample of the CAR. Percent sulfur (wet basis) is given by the following formula

Percent Sulfur (wet basis)

$$= \frac{(\text{volume of KIO3 consumed})(\text{furnace constant})}{\text{Mass of sample}}$$

where,

furnace constant = 1.1113

and  $\text{KIO}_3$  = potassium iodate

Putting in values into the above formula we get

$$\text{Percent sulfur (wet Basis)} = \frac{(0.035)(1.1113)}{(0.0573)} = 0.679 \%$$

Average percent sulfur content (dry basis) was found by the following formula

Average percent sulfur (dry basis)

$$= \frac{\text{Average percent sulfur (wet basis)}}{(100 - \text{percent moisture})} \times 100$$

Putting in values in the above formula we get

$$\text{Percent sulfur (dry basis)} = \frac{0.738 \times 100}{(100 - 28.83)} = 1.04 \%$$



## Appendix G

### MATERIAL BALANCE CLOSURE, REMOVAL AND NORMALIZATION FOR SULFUR

Total sulfur going into the process must come out in some form or another. Total amount of sulfur going into the process equals total amount of sulfur in the CAR processed. Total amount of sulfur coming out of the process equals total amount of sulfur in the hot water dried coal plus total amount of sulfur in the filtrate plus total amount of sulfur in the gas formed.

The sulfur was present in the gas as hydrogen sulfide, and the hydrogen sulfide content of gas was negligible and in most cases undetectable. Hence, the sulfur present in gas was neglected for the purpose of sulfur material balance closures.

#### SAMPLE DATA FOR RUN #5

- (i) Mean particle diameter= 0.4699 cm
- (ii) Temperature of run = 296 C
- (iii) Slurry concentration = 1:1 (solid to liquid mass ratio)
- (iv) Amount of filtrate obtained = 141.11 g
- (v) Amount of CAR processed (wet) = 146 g
- (vi) Amount of HWD coal obtained (wet) = 108.5 g

(vii) Sulfur content (wet) in CAR = 0.354 %

(viii) Sulfur content (wet) in hwd coal = 0.353 %

(ix) Sulfur content in filtrate = 0.048 %

Also,

Amount of sulfur in CAR = 0.5168 g

Amount of sulfur in HWD coal = 0.3830 g

Amount of sulfur in filtrate = 0.0677 g

#### SAMPLE MATERIAL BALANCE CLOSURE FOR SULFUR

Percent sulfur closure

$$= (A + B)(100)/C$$

where,

A = Total amount of sulfur in HWD coal, g

B = Total amount of sulfur in filtrate, g

C = Total amount of sulfur in CAR, g

Substituting the values we get,

$$\begin{aligned}\text{Percent sulfur closure} &= (0.3830 + 0.0677)(100)/0.51688 \\ &= 87.2 \%\end{aligned}$$

#### SAMPLE CALCULATION FOR SULFUR REMOVAL

Percent sulfur removal (HWD coal basis)

$$= (M - N)(100)/M$$

where,

M = Total amount of sulfur in CAR, g

N = total amount of sulfur in HWD coal, g

Substituting the values we get,

Percent sulfur removal (HWD coal basis)

$$= (0.5168 - 0.3830)(100)/0.5168$$

$$= 25.90 \%$$

Percent sulfur removal (filtrate basis)

$$= \frac{(\text{Total sulfur in filtrate})(100)}{\text{Total sulfur in CAR}}$$

$$= (0.0677)(100)/0.5168$$

$$= 13.20 \%$$

#### NORMALIZATION FOR SULFUR

Normalized amount of Sulfur in HWD coal

$$= \frac{\text{Amount of Sulfur in HWD coal (determined)}}{\text{Sulfur closure}}$$

$$= \frac{0.383\text{g}}{0.87} = 0.440\text{g}$$

Similarly, normalised amount of sulfur in filtrate

$$= \frac{\text{Amount of sulfur in filtrate (determined)}}{\text{Sulfur closure}}$$

$$= \frac{0.067\text{g}}{0.87} = 0.078 \text{ g}$$

Normalised percent sulfur removal

$$= \frac{\text{Percent sulfur removal (filtrate basis)}}{\text{Sulfur closure}}$$

Therefore,

Normalised percent sulfur removal

$$= 13.20 \%/0.87$$

$$= 15.10 \%$$



## Appendix H

### CALCULATION OF SODIUM, POTASSIUM, MAGNESIUM, AND CALCIUM CONTENTS OF THE COAL AND THE FILTRATE

The following is a sample calculation for the various metal contents in a sample of coal and filtrate.

#### SAMPLE DATA FOR RUN #19

1. Temperature of drying = 262.5 C
2. Mean particle diameter = 0.0505 cm
3. Type of coal = CAR
4. Weight of container = 3.1222 g
5. Weight of container and ash = 3.2369 g
6. Weight of ash = 0.1142 g
7. Average percent ash (dry) = 21.54 %
8. Average percent moisture = 28.83 %

The readings off the atomic absorption spectrophotometer were :

The ppm for sodium

For 1 dilution CAR solution = 10 ppm

The ppm for potassium

For 10 dilution CAR solution = 1.02 ppm

The ppm for magnesium

For 100 dilution CAR solution = 0.15 ppm

The ppm for calcium

For 100 dilution CAR solution = 1.00 ppm

SAMPLE CALCULATION FOR THE METALS CONTENT

From these readings, the metals content were calculated as follows.

For sodium

The sodium content in the CAR

$$\begin{aligned}
 &= \frac{10 \text{ g Na}}{10^6 \text{ g solution}} \times 100 \text{ ml solution} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{21.54 \text{ g ash}}{(100-21.54) \text{ g MAF ash}} \times 1 \text{ dil} \\
 &= 0.0024 \frac{\text{g Na}}{\text{g MAF coal}}
 \end{aligned}$$

For potassium

The potassium content in the CAR

$$\begin{aligned}
 &= \frac{1.02 \text{ g K}}{10^6 \text{ g solution}} \times 100 \text{ ml solution} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{21.54 \text{ g ash}}{(100-21.54) \text{ g MAF ash}} \times 10 \text{ dil} \\
 &= 0.0024 \frac{\text{g K}}{\text{g MAF coal}}
 \end{aligned}$$

For magnesium

The magnesium content in the CAR

$$\begin{aligned}
 &= \frac{0.15 \text{ g Mg}}{10^6 \text{ g solution}} \times 100 \text{ ml solution} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{21.54 \text{ g ash}}{(100-21.54) \text{ g MAF ash}} \times 100 \text{ dil} \\
 &= 0.0036 \frac{\text{g Mg}}{\text{g MAF coal}}
 \end{aligned}$$

For calcium

The calcium content in the CAR

$$\begin{aligned}
 &= \frac{1.00 \text{ g Ca}}{10^6 \text{ g solution}} \times 100 \text{ ml solution} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{21.54 \text{ g ash}}{(100-21.54) \text{ g MAF ash}} \times 100 \text{ dil}
 \end{aligned}$$

$$= 0.0243 \frac{\text{g Ca}}{\text{g MAF coal}}$$

The 'dil' in the above formulae refer to 'dilution'. The same procedure was followed for calculating the metals content of the hot water dried coal.

#### SAMPLE CALCULATION OF THE METAL OXIDES CONTENT IN THE ASH

The following is a sample calculation of the metal oxides content in the ash of the CAR used for the run #19.

For sodium oxide

The sodium oxide content in the ash of the CAR

$$= \frac{10 \text{ g Na}}{10^6 \text{ g solution}} \times 100 \text{ ml} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{62 \text{ g Na}_2\text{O}}{46 \text{ g Na}} \times 100 \% \times 1 \text{ dil} = 1.18 \% \text{ Na}_2\text{O in ash}$$

For sodium oxide

The potassium oxide content in the ash of the CAR

$$= \frac{10 \text{ g K}}{10^6 \text{ g solution}} \times 100 \text{ ml} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{94 \text{ g K}_2\text{O}}{78 \text{ g K}} \times 100 \% \times 10 \text{ dil} = 10.76 \% \text{ K}_2\text{O in ash}$$

For magnesium oxide

The magnesium oxide content in the ash of the CAR

$$= \frac{0.15 \text{ g Mg}}{10^6 \text{ g solution}} \times 100 \text{ ml} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{40 \text{ g MgO}}{24 \text{ g Mg}} \times 100 \% \times 100 \text{ dil} = 2.19 \% \text{ MgO in ash}$$

For calcium oxide



The calcium oxide content in the ash of the CAR

$$= \frac{1.0 \text{ g Ca}}{10^6 \text{ g solution}} \times 100 \text{ ml solution} \times \frac{1}{0.1142 \text{ g ash pellet}} \times \frac{56 \text{ g CaO}}{40 \text{ g Ca}} \times 100 \% \times 100 \text{ dil} = 12.36 \% \text{ CaO in ash}$$

The metal oxide content of the hot water dried coal was determined by the same procedure.

#### SAMPLE DATA FOR THE METALS CONTENT OF THE FILTRATE

The following is a sample data for the metals content of the filtrate of the run #19.

The ppm for sodium

For 50 dilution filtrate solution = 20 ppm

The ppm for potassium

For 50 dilution filtrate solution = 0.12 ppm

The ppm for magnesium

For 400 dilution solution = 0.05 ppm

The ppm for calcium

For 400 dilution solution = 1.98 ppm

#### SAMPLE CALCULATION FOR THE METALS CONTENT OF THE FILTRATE

A sample calculation for the metals content of the filtrate is as follows.

The amount of sodium present in the filtrate

$$= (20)(50 \text{ dil})(10^{-6}) = 1000 \frac{\text{g Na}}{\text{ml filtrate}}$$

The amount of potassium present in the filtrate

$$= (0.12)(50 \text{ dil})(10^{-6}) = 6.0 \frac{\text{g K}}{\text{ml filtrate}}$$

The amount of magnesium present in the filtrate

$$= (0.05)(400 \text{ dil})(10^{-6}) = 20 \frac{\text{g Mg}}{\text{ml filtrate}}$$

The amount of calcium present in the filtrate

$$= (1.98)(400 \text{ dil})(10^{-6}) = 794 \frac{\text{g Ca}}{\text{ml filtrate}}$$

## Appendix I

### MATERIAL BALANCE CLOSURE, REDUCTION AND NORMALIZATION FOR METALS

The amount of metals going into the process must come out in some form or the other. Total amount of metals going into the process equals the total amount of metals in the CAR processed. Total amount of metals coming out of the process equals the total amount of metals in the hot water dried coal plus the total amount of metals in the filtrate. These metals refer to sodium, potassium, magnesium, and calcium.

#### SAMPLE DATA FOR RUN #11

1. Temperature of run = 360 C
2. Particle size = 0.4699 cm
3. Slurry concentration = 1:1(solid to liquid mass ratio)
4. Amount of MAF coal in CAR = 92.5 g
5. Amount of MAF coal in HWD coal = 76.6 g
6. Amount of filtrate obtained = 161 ml

The metal contents of the CAR, the HWD coal, and the filtrate were as follows :

1. Sodium content of CAR = 0.0027 g Na/g MAF coal
2. Potassium content in CAR = 0.0013 g K/g MAF coal



3. Magnesium content in CAR = 0.0026 g Mg/g MAF coal
4. Calcium content in CAR = 0.0199 g Ca/g MAF coal
5. Sodium content in HWD coal = 0.00063 g Na/g MAF coal
6. Potassium content in HWD coal = 0.0012 g K/g MAF coal
7. Magnesium content in HWD coal = 0.0031 g Mg/g MAF coal
8. Calcium content in HWD coal = 0.0237 g Ca/g MAF coal
9. Sodium content in filtrate = 0.0008 g Na/ml filtrate
10. Potassium content in filtrate = 0.000037 g K/ml filtrate
11. Magnesium content in filtrate = 0.000012 g Mg/ml filtrate
12. Calcium content in filtrate = 0.00112 g Ca/ml filtrate

SAMPLE CALCULATION FOR MATERIAL BALANCE CLOSURE FOR SODIUM

Total Na in CAR = 0.24975 g

Total Na in HWD coal = 0.04826 g

Total Na in filtrate = 0.12880 g

Percent sodium closure

$$\begin{aligned}
 &= \frac{(\text{Total Na in HWD coal and filtrate})(100)}{\text{Total Na in CAR}} \\
 &= (0.17706)(100)/0.24975 \\
 &= 71 \%
 \end{aligned}$$

SAMPLE CALCULATION FOR THE REMOVAL OF SODIUM

Percent sodium removal (HWD coal basis)

$$\begin{aligned}
 &= \frac{(\text{Total Na in CAR} - \text{Total Na in HWD coal})(100)}{\text{Total Na in CAR}} \\
 &= (0.24975 - 0.04826)(100)/0.24975 \\
 &= 80.70 \%
 \end{aligned}$$

Percent sodium removal (filtrate basis)

$$\begin{aligned}
 &= \frac{(\text{Total Na in filtrate})(100)}{\text{Total Na in CAR}} \\
 &= (0.12880)(100)/0.24975 \\
 &= 51.6 \%
 \end{aligned}$$

NORMALIZATION FOR SODIUM

Normalized amount of sodium in HWD coal

$$\begin{aligned}
 &= \frac{\text{Amount of Na in HWD coal (determined)}}{\text{Na Closure}} \\
 &= \frac{0.04826 \text{ g}}{0.71} = 0.06797 \text{ g}
 \end{aligned}$$

Similarly, the normalized amount of sodium in the filtrate

$$\begin{aligned}
 &= \frac{\text{Amount of Na in the filtrate}}{\text{Na Closure}} \\
 &= \frac{0.1288 \text{ g}}{0.71} = 0.1814 \text{ g}
 \end{aligned}$$

Normalized percent sodium removal

$$\begin{aligned}
 &= \frac{\text{Percent Na removal (filtrate basis)}}{\text{Na closure}} \\
 &= 51.6 \%/0.71
 \end{aligned}$$

$$= 72.7 \%$$

SAMPLE MATERIAL BALANCE CLOSURE FOR POTASSIUM

$$\text{Total K in CAR} = 0.12025 \text{ g}$$

$$\text{Total K in CAR} = 0.09192 \text{ g}$$

$$\text{Total K in filtrate} = 0.005957 \text{ g}$$

Percent K closure

$$\begin{aligned} &= \frac{(\text{Total K in HWD coal and filtrate})(100)}{\text{Total K in CAR}} \\ &= (0.09192 + 0.00596)(100) / 0.12025 \\ &= 81.40 \% \end{aligned}$$

SAMPLE CALCULATION FOR THE REMOVAL OF POTASSIUM

Percent potassium removal (HWD coal basis)

$$\begin{aligned} &= \frac{(\text{Total K in CAR} - \text{Total K in hwd coal})(100)}{\text{Total K in CAR}} \\ &= (0.12025 - 0.09192)(100) / 0.12025 \\ &= 23.50 \% \end{aligned}$$

Percent potassium removal (filtrate basis)

$$\begin{aligned} &= \frac{(\text{Total K in filtrate})(100)}{\text{Total K in CAR}} \\ &= (0.00596)(100) / 0.12025 \\ &= 5 \% \end{aligned}$$



NORMALIZATION FOR POTASSIUM

Normalized amount of potassium in the HWD coal

$$\begin{aligned}
 &= \frac{\text{Amount of K in the HWD coal}}{\text{K Closure}} \\
 &= \frac{0.09192 \text{ g}}{0.81} = 0.1129 \text{ g}
 \end{aligned}$$

Similarly, the normalized amount of potassium in the filtrate

$$\begin{aligned}
 &= \frac{\text{Amount of K in the filtrate}}{\text{K Closure}} \\
 &= \frac{0.005957 \text{ g}}{0.81} = 0.007318 \text{ g}
 \end{aligned}$$

Normalized percent potassium removal

$$\begin{aligned}
 &= \frac{\text{Percent K removal (filtrate basis)}}{\text{K closure}} \\
 &= 5 \% / 0.814 \\
 &= 6.14 \%
 \end{aligned}$$

SAMPLE DATA FOR MAGNESIUM

Total Mg in CAR = 0.24420 g

Total Mg in HWD coal = 0.23976 g

Total Mg in filtrate = 0.00193 g

Percent magnesium closure

$$\begin{aligned}
 &= \frac{(\text{Total Mg in HWD coal and filtrate})(100)}{\text{Total Mg in CAR}} \\
 &= (0.23976 + 0.00193)(100) / 0.24420 \\
 &= 99 \%
 \end{aligned}$$

SAMPLE CALCULATION FOR THE REMOVAL OF MAGNESIUM

Percent magnesium removal (HWD coal basis)

$$\begin{aligned}
 &= \frac{(\text{Total Mg in CAR} - \text{Total Mg in HWD coal})(100)}{\text{Total Mg in CAR}} \\
 &= (0.24420 - 0.23976)(100) / 0.24420 \\
 &= 1.8 \%
 \end{aligned}$$

Percent magnesium removal (filtrate basis)

$$\begin{aligned}
 &= \frac{(\text{Total Mg in filtrate})(100)}{\text{Total Mg in CAR}} \\
 &= (0.01932)(100) / 0.24420 \\
 &= 0.8 \%
 \end{aligned}$$

NORMALIZATION FOR MAGNESIUM

The normalized amount of magnesium in the HWD coal

$$\begin{aligned}
 &= \frac{\text{Amount of Mg in the HWD coal}}{\text{Mg Closure}} \\
 &= \frac{0.2398 \text{ g}}{0.99} = 0.2422 \text{ g}
 \end{aligned}$$

Similarly, the normalized amount of magnesium in the filtrate

$$\begin{aligned}
 &= \frac{\text{Amount of Mg in the filtrate}}{\text{Mg Closure}} \\
 &= \frac{0.001932 \text{ g}}{0.99} = 0.001952 \text{ g}
 \end{aligned}$$

Normalized percent magnesium removal

$$\begin{aligned}
 &= \frac{\text{Percent Mg removal (filtrate basis)}}{\text{Mg closure}}
 \end{aligned}$$

$$= 0.8 \text{ \%}/0.99$$

$$= 0.81 \text{ \%}$$

#### SAMPLE DATA FOR CALCIUM

$$\text{Total Ca in CAR} = 1.8407 \text{ g}$$

$$\text{Total Ca in HWD coal} = 1.8154 \text{ g}$$

$$\text{Total ca in filtrate} = 1.8032 \text{ g}$$

Percent calcium closure

$$\begin{aligned} &= \frac{(\text{Total Ca in HWD coal and filtrate})(100)}{\text{Total Ca in CAR}} \\ &= (1.8154 + 0.18032)(100)/1.8407 \\ &= 108 \text{ \%} \end{aligned}$$

#### SAMPLE CALCULATION FOR THE REMOVAL OF CALCIUM

Percent calcium removal (HWD coal basis)

$$\begin{aligned} &= \frac{(\text{Total Ca in CAR} - \text{Total Ca in HWD coal})(100)}{\text{Total Ca in CAR}} \\ &= (1.8407 - 1.8154)(100)/1.8407 \\ &= 1.3 \text{ \%} \end{aligned}$$

Percent Ca removal (filtrate basis)

$$\begin{aligned} &= \frac{(\text{Total Ca in filtrate})(100)}{\text{Total Ca in CAR}} \\ &= (0.18032)(100)/1.8407 \\ &= 9.8 \text{ \%} \end{aligned}$$

NORMALIZATION FOR CALCIUM

The normalized amount of calcium in the hot water dried coal

$$\begin{aligned}
 &= \frac{\text{The amount of calcium in the HWD coal}}{\text{Ca closure}} \\
 &= \frac{1.8154 \text{ g}}{1.08} = 1.6809 \text{ g}
 \end{aligned}$$

Similarly, the normalized amount of calcium in the filtrate

$$\begin{aligned}
 &= \frac{\text{The amount of calcium in the filtrate}}{\text{Ca closure}} \\
 &= \frac{0.1803 \text{ g}}{1.08} = 0.1669 \text{ g}
 \end{aligned}$$

Normalized percent calcium removal

$$\begin{aligned}
 &= \frac{\text{Percent Ca removal (filtrate basis)}}{\text{Ca closure}} \\
 &= 9.80\%/1.08 \\
 &= 9.08 \%
 \end{aligned}$$



## Appendix J

### DETERMINATION OF TOTAL ACID GROUPS CONTENT IN COAL AND REDUCTION IN ACID GROUPS CONTENT ON HOT WATER DRYING

The following reagents were used for the determination of total acid groups content in CAR and HWD coal

1. Hydrochloric Acid,  $\text{HCl}$ , approximately 1.024 N
2. Barium Hydroxide,  $\text{Ba}(\text{OH})_2$ , approximately 0.3108 N

The pH-meter used for the potentiometric titration was Fisher Accumet model 230A pH/ion meter. The following is a sample calculation for acid groups content of a sample of coal and acid group reduction due to hot water drying.

#### SAMPLE DATA FOR RUN #8

1. Temperature of run = 311 C
2. Type of coal: CAR
3. Amount of CAR processed = 147 g
4. Mass of sample CAR = 5.7474 g
5. Percent moisture in CAR = 23.60 %
6. Volume of  $\text{Ba}(\text{OH})_2$  added to the coal samples initially = 100 cc
7. Reaction time (stirring without heat) = 24 hr

SAMPLE CALCULATION FOR ACID GROUPS CONTENTS

The following procedure was adopted to determine the acid groups content of a sample of CAR used for run #5. After 24 hrs of reaction between the coal sample and  $\text{Ba}(\text{OH})_2$ , the solution was filtered and a potentiometric titration was carried out on the filtrate. The volume of  $\text{HCl}$  added to the filtrate and the corresponding pH were noted. The titration was continued till the pH of the filtrate fell to about 2.3.

From the volume of  $\text{HCl}$  versus pH data, a neutralization curve was drawn. The volume of  $\text{HCl}$  required to reach the neutral point was found out by completing the parallelogram along the straight line section of the neutralization curve. The neutral point, or the equivalence point, is the point on the curve where the diagonals intersect. The equivalence point for the CAR is 13.6 ml  $\text{HCl}$ .

The amount of 0.3108N  $\text{Ba}(\text{OH})_2$  that reacted with 13.6 ml of 1.024 N  $\text{HCl}$  was found as follows:

$$(\text{Normality})(\text{Volume of Ba}(\text{OH})_2) = (\text{Normality})(\text{Volume of HCl})$$

$$(0.3108)(\text{Vol. of Ba}(\text{OH})_2) = (1.024)(13.6)$$

Therefore, the volume of  $\text{Ba}(\text{OH})_2 = 44.81$  ml

This is also the amount of  $\text{Ba}(\text{OH})_2$  that did not react with the acid groups in the coal. Hence,

Volume of  $\text{Ba}(\text{OH})_2$  that reacted with the acid groups

$$= 100 \text{ ml} - (\text{volume of Ba}(\text{OH})_2 \text{ that did not react})$$

$$= (100 - 44.81) \text{ ml} = 55.19 \text{ ml}$$

The amount of  $\text{Ba}(\text{OH})_2$  that reacted with the acid groups is given by

$$\begin{aligned} \text{Milliequivalents of } \text{Ba}(\text{OH})_2 &= (55.19 \text{ ml})(0.3108 \text{ N}) \\ &= 17.15 \text{ meq} \end{aligned}$$

This is also the amount of acid groups present in the CAR sample.

$$\begin{aligned} \text{Milliequivalents of acid/g of wet CAR} &= 17.15 \text{ meq}/5.7474 \text{ g of wet CAR} \\ &= 2.984 \text{ meq/g of wet CAR} \end{aligned}$$

And,

$$\begin{aligned} \text{Milliequivalents of acid/g of dry CAR} &= 17.15 \text{ meq}/4.3910 \text{ g of dry CAR} \\ &= 3.906 \text{ meq/g of dry coal} \end{aligned}$$

$$\begin{aligned} \text{Total amount of acid groups present in the CAR processed} &= (2.984 \text{ meq/g wet CAR})(147 \text{ g wet CAR}) \\ &= 439 \text{ meq} \end{aligned}$$

Similar calculations were done for the determination of acid groups content of the hot water dried coal.

The total amount of acid groups present in the hot water dried coal obtained = 125 meq

SAMPLE CALCULATION FOR REDUCTION IN ACID GROUPS CONTENT

The percent reduction in acid groups content on hot water drying

$$= \frac{(\text{Total acid in CAR} - \text{Total acid in HWD coal})(100)}{\text{Total acid groups in CAR}}$$

Therefore,

Percent reduction of acid groups

$$= (439 - 125)100/439 = 71.50\%$$



## Appendix K

### CARBON DIOXIDE CONTENT OF THE RESIDUAL GAS

The carbon dioxide content of the residual gas was calculated because it was related to amount of carboxylic acid groups decomposed during the process. The residual gas and its constituent gases were assumed to behave like ideal gases and hence, assumed to obey ideal gas laws.

The ideal gas law assumed to apply was

$$PV = nRT$$

where,

P = absolute pressure in the autoclave, Kg/m<sup>2</sup>

V = volume occupied by the gas in the autoclave, m<sup>3</sup>

n = number of moles of gas in the autoclave, Kg/mole

R = universal gas constant, (m)(Kg)/(Kg/mole)(K)

T = temperature in the autoclave, K

#### SAMPLE DATA FOR RUN #11

Absolute pressure in the autoclave, P = 157931 Kg/m<sup>2</sup>

Temperature in the autoclave, T = 305 K

Universal gas constant, R = 848 (m)(Kg)/(Kg/mole)(A)

Volume occupied by the gas in the autoclave

= (total available empty space in the autoclave)

- (space occupied by the coal slurry)

$$= (500.5 - 280) \text{ ml}$$

$$= 220.5 \text{ ml} = 0.0002205 \text{ cu. m}$$

Mole fraction of CO<sub>2</sub> in the residual gas = 0.8080

SAMPLE CALCULATION FOR CARBON DIOXIDE CONTENT OF RESIDUAL GAS

Now, substituting these values in the above gas law equation we get,

$$(157931 \text{ Kg/m}^2)(0.0002205 \text{ m}^3) = (n)(848 \text{ mKg/Kgmole K})(305 \text{ K})$$

which after solving for n gives,

$$n = 0.00013464 \text{ kgmole}$$

or,

$$n = 0.135 \text{ gmole}$$

This is the total amount of residual gas formed in the autoclave during the process. Therefore, the moles of CO<sub>2</sub> present in the residual gas is given by

$$\text{Moles of CO}_2 = (\text{moles of residual gas})(\text{mole fraction of CO}_2)$$

Or,

$$\begin{aligned} \text{Moles of CO}_2 &= (0.135 \text{ gmole})(0.808) \\ &= 0.109 \text{ gmoles} \end{aligned}$$

This is the CO<sub>2</sub> formed from 145.6 g of the original coal processed. Therefore the amount of CO<sub>2</sub> formed per 100 g of the CAR is given by

$$\begin{aligned} &= (0.109 \text{ g moles CO}_2)(100)/145.6 \text{ g CAR} \\ &= 0.075 \text{ g moles of CO}_2 \text{ per 100 g CAR} \end{aligned}$$

Appendix L  
CALORIFIC VALUE OF COAL

Calorific values of CAR and hot water dried coals were determined to find out the effect of drying temperature on the calorific value of coal. An adiabatic calorimeter was used for the determination of the calorific value. A sample calculation for the calorific value of a sample of CAR is shown below. Calorific value of the HWD coal is determined in a similar procedure.

SAMPLE DATA FOR RUN #5

1. Type of coal : CAR
2. Mean particle diameter = 0.4699 cm
3. Moisture content = 25.18 %
4. Sulfur content (wet basis) = 0.354%
5. Mass of container = 12.1873 g
6. Mass of container + sample = 13.1660 g
7. Therefore, the mass of the sample = 0.9787 g
8. Initial temperature of calorimeter = 22.041 C
9. Final temperature of calorimeter = 23.915 C
10. Therefore, the temperature rise = 1.874 C

CORRECTIONS TO BE MADE

The following corrections were made for the determination of the gross calorific value of the coal. The fuse wire correction is due to the combustion of the fuse wire, and the acid correction is due to the formation of sulfuric and nitric acids. The acid correction accounts totally for the heat of formation of nitric acid and and partially accounts for the heat of formation of sulfuric acid. The remaining part of the heat of formation of sulfuric acid is accounted for by the correction for the heat of formation of sulfuric acid shown below.

An iron alloy fuse wire was used to burn the sample coal in the calorimeter bomb. The fuse wire had a calorific value of 1240 cal/cm. From the length of the wire remaining after the coal combustion, the wire correction is found out by the following formula

$$\text{Wire correction} = (M - N)(1240 \text{ Cal/cm})$$

where,

1240 = Calorific value of fuse wire

M = Initial length of wire, cm

N = Final length of wire, cm

Here,

$$M = 10.2 \text{ cm}$$

$$N = 4.1 \text{ cm}$$

Substituting the values into the formula we get,

$$\text{Wire correction} = (10.2 \text{ cm} - 4.1 \text{ cm})(1240 \text{ cal/cm})$$



$$= 7560 \text{ cal}$$

Water present in the bomb after combustion of the coal sample, which contains dissolved acid fumes, was collected into a clean dry beaker and tritrated against a standard alkali (sodium carbonate of 0.1305 N). The 'ml' of alkali consumed till the end point directly gave the acid correction.

Here,

$$\text{Acid correction} = 1562 \text{ cal}$$

The water equivalent specification of the  
calorimeter = 10,96200 cal/C

#### SAMPLE CALCULATION FOR THE CALORIFIC VALUE

The following procedure is adopted to calculate the calorific value.

The correction for the heat of formation of sulfuric acid

$$\begin{aligned} &= (252)(23)(\% \text{ sulfur, wet basis})(\text{mass of sample, g}) \\ &= (252)(23)(0.354)(0.978) \\ &= 2008 \text{ cal} \end{aligned}$$

The gross heat of combustion, or calorific value,

$$= \frac{(0.0022)(\text{wat. equiv. of cal.})(\text{temp. rise}) - (\text{corr.})}{(\text{weight of the sample, g})}$$

Substituting the values into the equation we get,

$$\text{Hg} = \frac{(0.0022)(10,96200)(1.874) - (7560 + 1562 + 2008)}{0.9787}$$

or,

$$= 4,593 \text{ cal/g (wet coal)}$$

or, on a dry basis,

$$= 6,138 \text{ cal/g (dry coal)}$$

# Appendix M

## ANALYSIS OF THE AS RECIEVED COAL

Table 27 and 28 below presents the analyses of the CAR used for each group of runs. They present only the most important analyses of the CAR. Table 27 presents the moisture content, the ash content, the sulfur content and the acid groups content and Table 28 presents the metals content.

TABLE 27

### Analysis of CAR

Run No.	Moisture Content, %	Ash Content(dry basis), %	Sulfur Content(dry basis), %	Acid Content, meq/g dry CAR
4	23.90	10.20	0.516	3.91
5,6	25.18	13.14	0.473	4.15
7,8	23.60	11.90	0.470	3.91
9	27.80	12.43	0.650	5.30
10,11	26.87	13.11	0.424	4.76
12,13,14	26.94	18.60	1.09	3.68
15,16,17	28.84	21.59	1.37	3.50
18,19,20	28.83	21.54	1.04	3.45

TABLE 28

## Analysis of CAR

Run No.	Sodium Content, g/g MAF CAR	Potassium Content, g/g MAF CAR	Magnesium Content, g/g MAF CAR	Calcium Content, g/g MAF CAR
4	0.0026	0.0008	0.0018	0.0159
5,6	0.0028	0.0010	0.0027	0.0197
7,8	0.0030	0.0009	0.0033	0.0269
9	0.0023	0.0010	0.0024	0.0171
10,11	0.0027	0.0013	0.0026	0.0199
12,13,14	0.0026	0.0027	0.0037	0.0232
15,16,17	0.0026	0.0032	0.0034	0.0287
18,19,20	0.0024	0.0024	0.0036	0.0242



Appendix N  
STATISTICAL ANALYSIS

The following statistical analyses were done on the results of the present study.

1. Test on the difference of the two mean percent moisture reductions for the two particle sizes.
2. Test on the mean percent ash of the HWD coal for both the particle sizes.
3. Test on the mean percent sulfur removal for both the particle sizes.

TEST ON THE DIFFERENCE OF THE TWO MEAN PERCENT MOISTURE REDUCTIONS

This test refers to the simple t-test on the difference between the two mean percent moisture reductions for the two particle sizes. Two assumptions are made in the following tests, viz,

1. The moisture reduction values are randomly distributed.
2. The population variances of the two particle sizes, though unknown, can be assumed to be equal.

The test statistic is

$$T = \frac{M - N}{S_p \sqrt{(1/m + 1/n)}}$$

with degrees of freedom,  $v = m + n - 2$

where

$$S_p^2 = \text{pooled variance}$$

Pooled variance is given by

$$S_p^2 = \frac{(m-1)S_1^2 + (n-1)S_2^2}{m+n-2}$$

$m, n$  = no. of observation for the first and  
second particle sizes respectively

$M, N$  = sample means for the first and the  
second particle sizes respectively

Null Hypothesis:  $H_0$

$$u_1 - u_2 = 0 \text{ (the means are the same)}$$

Alternate Hypothesis :  $H_A$

$$u_1 - u_2 \neq 0 \text{ (the means are not the same)}$$

The t-test to be performed is a two tailed test, and hence  
the rejection region for the null hypothesis is

$$T > t(v, 1 - \alpha/2) \quad \text{or}$$

$$T < -t(v, 1 - \alpha/2)$$

where,

$u_1, u_2$  = population means for the moisture reduction  
for the first and second particle sizes  
respectively

$\alpha$  = significance level of the test

' $t(v, 1 - \alpha/2)$ ' is found from the statistical tables (37).

For the 0.4699 cm mean diameter particles,

$$\text{Mean} = \frac{(\text{Summation of the moisture reductions})}{(\text{No. of observations})}$$

Substituting the values into the above formula we get

$$M = \frac{(75.11+72.64+75.58+70.43+75.85+73.06+70.40+67.13)}{8} = 72.52 \%$$

Also the sample variance,

$$S_1^2 = 9.3$$

$$m = 8$$

Similarly for the 0.0505 cm mean diameter particles,

$$N = \frac{(60.22+66.51+74.29+76.90+77.40+82.32+90.81+85.31+81.57)}{9} = 77.25 \%$$

Sample variance,

$$S_2^2 = 88.43$$

$$\text{and } n = 9$$

Substituting the values into the formula for pooled variance we get

$$S_p^2 = \frac{(7)(9.3)+(8)(88.43)}{15} = 51.5$$

Substituting the values into the formula for T we get

$$T = \frac{72.52 - 77.25}{\sqrt{(51)(1/8+1/9)}} = -1.36$$

Let the level of significance of the test be,

$$a = 5 \%$$

We have,

$$v = m + n - 2 = 15$$

From the t-tables we get,

$$t(15, 0.975) = 2.131$$

$$-t(15, 0.975) = -2.131$$

Therefore we find that,

$$-t(15, 0.975) < T < t(15, 0.975)$$

Therefore accept the null hypothesis,

That is,

$$u_1 - u_2 = 0$$

There is no significant difference between the two mean percent moisture reductions for the two particle sizes. The moisture reductions for the two particle sizes is significantly the same, and hence there is no significant effect of particle size on moisture reduction.

#### TEST ON THE MEAN PERCENT ASH OF THE HOT WATER DRIED COAL

The test performed determines whether a random sample of percent ash of hot water dried coal of a particular size comes from a population of ash contents of the CAR with mean  $u_0$ . Or, in other words, this test determines whether the populations of ash of hot water dried coal and CAR are the same or not. The test is performed for both particle sizes. Random distribution of percent ash is assumed for both the particle sizes.



Since the population variance is unknown, the test statistic is

$$T = \frac{M - u_0}{S/\sqrt{(n)}}$$

and the degrees of freedom,  $v = n - 1$

where,

$M$  is the sample mean

$u_0$  is the population mean

$S^2$  is the sample variance

and  $n$  is the sample size

Let the significance level desired be,  $\alpha = 5\%$

Null Hypothesis:  $H_0$

$\mu = \mu_0$ , the ash population means of HWD coal and  
CAR are the same

Alternate Hypothesis:  $H_A$

$\mu \neq \mu_0$ , the ash population means of HWD coal and  
CAR are not the same.

The test performed is a two tailed test. Hence the rejection region for the null hypothesis is

$$T > t(v, 1 - \alpha/2)$$

or 
$$T < -t(v, 1 - \alpha/2)$$

For the 0.4699 cm mean diameter particles,

$$M = \frac{(15.30+11.24+12.49+13.50+12.60+12.69+14.90+13.93)}{8} = 13.33\%$$

Sample variance,

$$S^2 = 1.35$$

Population mean,

$$u_0 = 12.37$$

And sample size,

$$n = 8$$

Substituting values into the formula for T we get,

$$T = \frac{13.33 - 12.37}{\sqrt{(1.35/8)}} = 2.337$$

From the tables we have,

$$t(7, 0.975) = 2.365$$

$$-t(7, 0.975) = -2.365$$

Hence we have from the above values,

$$-t(7, 0.975) < T < t(7, 0.975)$$

Therefore, accept the null hypothesis,

$$u = u_0$$

That is, there is no difference in the ash contents of the CAR and the hot water dried coals for the 0.4699 cm mean diameter particles.

Similarly for the 0.0505 cm mean diameter particles,

$$M = \frac{(19.65+18.61+19.23+21.73+20.81+22.44+21.76+20.82+22.45)}{9} = 20.83$$

The sample variance, size and population mean are 1.404, 9 and 20.58 respectively.

Substituting values into the formula for T we get,

$$T = \frac{20.83 - 20.58}{\sqrt{(1.404/9)}} = 0.633$$

From the tables,

$$t(8, 0.975) = 2.306$$

$t(8, 0.975) = -2.306$  Hence we have from the above values,

$$-t(8, 0.975) < T < t(8, 0.975)$$

The above region is not in the rejection region for the null hypothesis. Therefore accept the null hypothesis,  $u = u_0$ . That is, there is no difference in ash contents of CAR and HWD coals for 0.0505 cm mean diameter particles.

#### TEST ON THE MEAN PERCENT SULFUR REMOVAL

The test performed determines whether a random sample of sulfur removal comes from a population with mean equal to 0. The test is performed for both particle sizes. A random distribution of percent sulfur removal is assumed for both the particle sizes.

Since the population variance is unknown, the test statistic is

$$T = \frac{M - u_0}{S/\sqrt{(n)}}$$

with the degrees of freedom,  $v = n - 1$

where,

$M$  is the sample mean

$u_0$  is the population mean

$S^2$  is the sample variance

and  $n$  is the sample size

Let the significance level desired be,  $\alpha = 5\%$

Null Hypothesis:  $H_0$

$\mu = \mu_0 (= 0)$ , the percent sulfur removal is  
equal to 0.

Alternate Hypothesis:  $H_A$

$\mu \neq \mu_0 (= 0)$ , the percent sulfur removal is  
not equal to 0.

The test performed is a two tailed test. Hence the rejection region for the null hypothesis is

$$T > t(v, 1-\alpha/2)$$

or 
$$T < -t(v, 1-\alpha/2)$$

For the 0.4699 cm mean diameter particles,

$$M = \frac{(14.80+15.20+7.40+10.40+0+7.52+7.33+10.90)}{8}$$

$$= 9.19\%$$

Sample variance,

$$s^2 = 4.87$$

Population mean,

$$\mu_0 = 0$$

And sample size,

$$n = 8$$

Substituting values into the formula for T we get,

$$T = \frac{9.19 - 0}{\sqrt{(4.87/8)}} = 11.77$$

From the tables,

$$t(7, 0.975) = 2.365$$

$$-t(7, 0.975) = -2.365$$



Hence from the above values,

$$T > t(7, 0.975)$$

The above region is rejection region for the null hypothesis. Therefore, reject the null hypothesis and accept the alternate hypothesis,  $u \neq 0$ .

That is, the sulfur removal is significantly different from 0, or, there is sulfur removal. Since the sulfur removal averages 9.19 percent, it is low.

Similarly for the 0.0505 cm mean diameter particles,

$$M = \frac{(2.79+3.08+5.57+6.97+8.23+6.49+4.94+7.57+5.18)}{9} = 5.65$$

Sample variance, size and population mean are 1.88, 9 and 0 respectively.

Substituting values into the formula for T we get,

$$T = \frac{5.65 - 0}{(1.880/9)} = 12.36$$

From the tables we have,

$$t(8, 0.975) = 2.306$$

$$t(8, 0.975) = -2.306$$

Hence we have from the above values,

$$T > t(8, 0.975)$$

The above region is in the rejection region for the null hypothesis. Therefore the alternate hypothesis,  $u \neq 0$  is accepted.

That is, the sulfur removal is significantly different from 0, or, there is sulfur removal. Sulfur removal averages 5.65 % which is low.

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